

EMACC ANNUAL TECHNICAL REPORT

FY 1976

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U.S. Energy Research and
Development Administration
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ANNUAL TECHNICAL REPORT

FY 1976

ERDA Materials Coordinating Committee

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MATERIALS SCIENCES

Division of Physical Research

Introduction:

The Materials Sciences subprogram of the Division of Physical Research supports basic materials research in energy related areas of interest To ERDA. While organizationally the Division of Physical Research is located under the Assistant Administrator for Solar, Geothermal and Advanced Energy Systems, the mission of the Division is to provide the physical research base for all ERDA activities. In addition to the Materials Sciences subprogram, other programs are administered by the Division's Nuclear Sciences, Molecular, Mathematical and Geo-sciences, and High Energy Physics Offices.

Materials Sciences research is supported primarily at ERDA National Laboratories and universities and to a lesser extent at industrial laboratories. The research covers a spectrum of scientific and engineering areas of interest to ERDA and is conducted generally by personnel trained in the disciplines of Solid State Physics, Metallurgy, Ceramics and Chemistry. The overall goals of the program are summarized in the following:

1. To advance the understanding of basic structures, mechanisms and phenomena governing properties and behavior of matter in the condensed state.
2. To provide a foundation for materials technology through the development of basic knowledge in Materials-related energy problem areas of interest ~~to ERDA~~.
3. To exploit the unique capabilities and facilities existing in ERDA Laboratories for conducting national materials sciences programs.

Most of the research is conducted at the ERDA multiprogram laboratories in close proximity to the applied programs. In this manner, the transfer of new information and techniques into technology is facilitated while at the same time the needs of the applied programs are brought to the attention of the basic research community. Special attention is also given to research capabilities residing in universities. Support of research in universities is justified on the basis of research opportunities at these institutions. Here, recognition is also given to the fact that highly trained manpower in the critical area of materials

science will result whose skills will be oriented toward energy technologies.

Funding for the Materials Sciences subprogram for FY 1976 was 43.9 M\$ in operating budget outlays. The research programs are described in considerable detail in the publication Materials Sciences Programs FY 1976 available as ERDA publication 76/123. The report also contains a convenient index to various subjects of interest.

Figure 1 shows the distribution of funding for the Materials Sciences program as planned for FY 1977, by laboratory. Note that the portion of the program entitled Contract Research is that which is made up of projects funded via the unsolicited proposal route and consists of mostly university projects. Figure 2 shows the distribution of funding by topical area.

In the following, a very brief description of materials sciences research is given by title and institution in two categories, Major Laboratories and Contract Research. Further information is available in ERDA 76/123. All funding is given in thousands of dollars, FY 1976 level.

Major Laboratories:

Ames Laboratory, (Gschneidner, Klierer, Corbett) \$4600

Research is underway on structure of materials, mechanical properties, physical properties, radiation effects, neutron scattering, magnetic properties of solids, nuclear resonance in solids, superconductivity, thermodynamic and transport properties of solids, optical and spectroscopic properties of solids and liquids, rare earth metals preparation, optical and surface physics theory, superconductivity theory, magnetic and electronic properties of solids theory, x-ray and neutron crystallography, low oxidation states in inorganic systems, chemistry of heavy transition metals, liquid metals, metals from fly ash, thermal emittance properties of materials, mass transfer and transport in fluids and particulate systems, high temperature chemistry and surface chemistry and catalysis.

Argonne National Laboratory, (Peterson, Price, Fields, Burns) \$10,700

Research is underway on alloy properties, scattering studies, physical metallurgy, properties of high-temperature MHD materials, catalysis and surface studies, mechanical properties-plasticity, mechanical

properties-erosion and wear, metal physics, superconductivity, charged-particle irradiation, neutron irradiation, kinetic studies, diffusion studies, neutron scattering studies, materials sciences research with the prototype pulsed neutron source, materials preparation and characterization, defects and impurities in non-metallic systems, low temperature studies, superconductivity studies, phase transitions and catalysis, magnetic properties, electronic properties, light scattering and acoustics, solar materials, refractory materials with MHD applications, solid state theory, particle solid interactions, neutron scattering and x-ray diffraction studies, physical and surface chemistry of energy systems, low temperature calorimetry, high temperature chemistry, liquid metals chemistry, molten salt chemistry, chemistry of materials, thermodynamic properties of inorganic substances, physical chemistry of electro-chemical energy storage and heat transfer materials and metastable fluids.

Brookhaven National Laboratory, (Gurinsky, Blume) \$4,700

Research is underway on superconductivity and relationship between properties and structures, radiation damage, neutron scattering-magnetic systems, neutron scattering-phase transitions, neutron scattering-elementary excitations in solids, neutron scattering-partially ordered systems, superconductivity, surface studies, low temperature physics, spectroscopy of solids, theoretical research, radiation effects research, properties of real solids and advanced materials synthesis and characterization.

Idaho National Engineering Laboratory, (Keiser) \$ 60

Research is underway on geothermal scaling and corrosion research.

University of Illinois, (Maurer) \$1,540

Research is underway on electronic structure and magnetism of transition metal alloys, dynamic structure of supercritical dense water and aqueous electrolyte solutions, physics of refractory materials, localized corrosion of passive metals, interstitial solid solutions, hydrogen behavior in BCC metals, applications of electron microscopy in materials science, deformation of reinforced metals, the mechanism of stress-corrosion cracking, precipitation in refractory metal alloys, dielectric solids, nuclear magnetic resonance studies of metals and polymers, physical and catalytic properties of catalysts, use of very high pressure to investigate the structure of matter, anharmonic effect in solids, defect properties of solids, properties of noble gas crystals, nuclear magnetic resonance in solids, radiation damage in solids, impurities in superconductors, response of solids to

electromagnetic radiation and low temperature studies of defects in solids.

Lawrence Berkeley Laboratory, (Shirley) \$3,890

Research is underway on microstructure properties and alloy design-electron diffraction and microscopy, powder metallurgy, theoretical problems in alloy design, fundamentals of alloy design, relations between dislocations, point defects and properties of crystalline materials, superconductivity effects-high field superconductivity, microstructure and mechanical behavior of ceramic materials: and glass-and ceramic-metal systems, high temperature reactions, relation of microstructure to properties in ceramics, structure and electrical properties of composite materials, far infrared spectroscopy, experimental solid state physics and quantum electronics, excited quantum fluids in solids, superconductivity, superconducting devices, and 1/f noise, theoretical solid state physics, high pressure chemistry, low-temperature properties of materials, mass and charge transport in electrochemical systems properties of nonaqueous ionizing solvents, high temperature thermodynamics, chemistry and materials problems in energy production technologies, crystallization kinetics, electrochemical phase boundaries, solid state and surface reaction studies, and nuclear magnetic resonance.

Lawrence Livermore Laboratory, (Roberts) \$ 320

Research is underway on hot corrosion studies related to fossil fuels, low index materials, optical coatings, and D₂-DT-T₂ phase diagram.

Low Alamos Scientific Laboratory, (Baker) \$ 610

Research is underway on high temperature materials for energy applications, high temperature neutron damage studies, CTR related chemical research tritium chemistry associated with the lithium blanket and container materials, and Los Alamos equation of state library.

Mound Laboratory, (Wittenberg) \$ 125

Research is underway on liquid metals research.

Oak Ridge National Laboratory, (McHargue, Wilkinson, Keller) \$10,640

Research is underway on ceramics research, preparation and synthesis of high temperature and special service materials, theory of the solid state, x-ray diffraction research, deformation and mechanical properties, kinetics and mechanisms of surface and solid state reactions, energy transport in solids, metallurgy of superconducting materials, radiation

effects, elementary excitations in condensed matter, magnetic properties of solids, properties of defects, superconductors, and hydrides, physical properties of ceramics, physical properties of superconductors, research and development on pure materials, surface studies and catalysis, photophysical processes of solar energy conversion, theory of condensed matter, low temperature radiation effects, x-ray diffraction and electron microscopy, ion bombardment, normalization of ion and neutron damage, chemical structure of energy-related materials, basic materials chemistry related to fusion reactor systems, thermodynamics and transport in molten salts and hydrous melts, surface chemistry, and electrochemical kinetics and corrosion.

Pacific Northwest Laboratory, (Nelson) \$ 830

Research is underway on ceramics for energy applications, structure-property relationships in sputter-deposited materials for solar applications, optical and laser material study, oxidation, corrosion, and wear resistant fine-grained materials, sputter-deposited superconductor research, transuranium physical metallurgy research and radiation effects on metals.

Sandia Laboratories, (Galt, Murphey) \$ 200

Research is underway on defects and impurities in ion-implanted insulators and semiconductors, surface physics research, and gases in metals.

Contract Research:

Arizona State University

Solid State Chemistry of Rare Earth Oxides, (Eyring) . \$ 55
Imaging Surfaces and Defects in Crystals, (Cowley) ... \$ 52
Study of Ferrite Formation in Neutron
Irradiated Austenitic Stainless Steels, (Stanley) .. \$ 29

Brown University

A Combined Macroscopic and Microscopic Approach
to the Fracture of Metals, (Gurland, Rice) \$ 85

California Institute of Technology

| | |
|---|-------|
| Studies of Alloy Structures and Properties, (Duwez) | \$170 |
| Metals Hydrides with Multiple Pulse Nuclear Magnetic Resonance Techniques, (Vaughan) | \$ 53 |

University of California/Los Angeles

| | |
|--|-------|
| High Temperature Irradiation Damage and Precipitation Hardening in NI-Base Alloys, (Ardell) | \$ 75 |
| Fourier Space Computer Simulation of Crystalline Imperfections, (de Fontaine) | \$ 39 |

University of California/Riverside

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|--|-------|
| Theoretical Aspects of Superconductor, (Simanek) | \$ 77 |
|--|-------|

University of California/San Diego

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|--|-------|
| The Response of Superconductors to Variations in Impurity Content and Applied Pressure, (Maple) | \$125 |
| Research on the Properties of Materials at Very Low Temperatures, (Wheatley) | \$214 |

University of California/Santa Barbara

| | |
|---|-------|
| Resonance Studies of Superionic Conductors, (Jaccarino).. | \$ 41 |
|---|-------|

Carnegie-Mellon University

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|--|-------|
| Generalization of Internal Centrifugal Zone Growth of Metal-Ceramic Composites, (Sekerka) | \$ 36 |
|--|-------|

Case Western Reserve University

| | |
|---|-------|
| Coupled Diffusion Phenomena in Multicomponent Glasses and Glass Forming Liquids, (Cooper) | \$ 52 |
| Dislocation-Solute Atom Interactions in Alloys, (Gibala) | \$ 53 |
| Experiments in High Voltage Electron Micro- scopy, (Mitchell) | \$ 77 |
| Elastic and Plastic Strains and the Stress Corrosion Cracking of Austenitic Stainless Steels, (Troiano) | \$ 39 |

University of Chicago

The Study of Phonons and Electronic Processes in
Ordered and Disordered Solids, (Solin) \$ 55

University of Cincinnati

Flux Pinning and Flux Flow Studies in Superconductors
Using Flux Flow Noise Techniques, (Joiner) \$ 39
Radiation Effects on BCC Refractory Metals and
Alloys, (Moteff) \$ 44

Clarkson College of Technology

Nucleation of Voids, (Katz) \$ 23

University of Colorado

Critical Scattering of Laser Light By Bulk Fluids
and Thin Fluid Films, (Mockler, O'Sullivan) \$ 59

Colorado School of Mines

Liquid Lithium Corrosion and Corrosion-Fatigue
Research, (Olson, Matlock) \$ 46

Columbia University

High Temperature Transport Properties and Processes
of Gases and Alkali Metals, (Bonilla) \$ 47

University of Connecticut

Electrode Polarization Studies in Hot
Corrosion Systems, (Devereux) \$ 41
Electron-Dislocation Interactions at Low
Temperatures, (Galligan) \$ 41
Cluster Carburizing, (Morral) \$ 39

Cornell University

Influence of Grain Boundaries on the Electrical Transport
Properties of Polycrystalline Si Film, (Ast) \$ 25
Structure and Properties of Grain Boundaries, (Balluffi) \$ 82
Reduction of Mixed Spinel Oxides, (DeJonghe) \$ 42
Environment and Fracture, (Johnson) \$ 59

Cornell University, continued

| | |
|--|-------|
| Theory of Structure and Dynamics in Condensed Matter, (Krumhansl) | \$120 |
| Mechanical Behavior of Materials and Structural Elements at Elevated Temperatures, (Lance) | \$ 60 |
| Mechanical Properties of Crystalline Solids, (Li) | \$ 63 |
| Probabilistic Models of the Stress-Rupture of Composite Materials, (Phoenix) | \$ 32 |
| Experimental Phonon Physics, (Pohl, Sievers) | \$250 |
| Defects in Metal Crystals, (Seidman) | \$180 |

Dartmouth College

| | |
|--|-------|
| Theory of Electron-Phonon Scattering Effects in Metals, (Lawrence) | \$ 28 |
| Experimental Determination of the Temperature Dependence of Metallic Work Functions at Low Temperatures, (Pipes) | \$ 28 |

Drexel University

| | |
|--|-------|
| Strain Hardening and Ductility of Iron: Axisymmetric vs. Plane Strain Elongation, (Langford) | \$ 43 |
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University of Florida

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|--|-------|
| Quantitative Analysis of Solute Segregation in Alloys by Transmission Electron Microscopy, (Hren, Hartley) | \$ 43 |
| Deformation Processes in Refractory Metals, (Reed-Hill) | \$ 40 |

Georgia Institute of Technology

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|--|-------|
| Investigations of Relationships Between Micro-structure, Magnetic Properties and the Hydriding Processes in Intermetallic Compounds of Rare Earth and Transition Metals, (Livesay) | \$ 60 |
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Georgetown University

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| The Study of Very Pure Metals At Low Temperatures, (Gregory) | \$ 70 |
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University of Hawaii

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| Pressure Derivatives of Elastic Moduli in BCC Transition Metals and their Solid Solutions, (Manghnani) | \$ 38 |
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University of Hawaii, continued

Photoelectric Emission from Thin Films in the Vacuum
Ultraviolet Region, (Pong) \$ 33

University of Houston

Microstructural Studies of Hydrogen and other Inter-
stitial Defects in BCC Refractory Metals, (Moss, McIntire) . \$ 54

Howard University

Radiation Damage in Optically Transparent
Materials (Zircons), (Thorpe) \$ 20

Illinois Institute of Technology

Thermal and Electrical Measurements on Solids at
Low Temperatures, (Weinstock) \$200

University of Kansas

High Temperature Chemistry, (Gilles) \$ 60

Lehigh University

Pressure Sintering and Creep Deformation-A Joint
Modeling Approach, (Notis) \$ 48

Marquette University

Defect Structures in Nonstoichiometric Oxides, (Blumenthal) .. \$ 69

University of Maryland

An Investigation of Irradiation Strengthening of BCC
Metals and Solid Solutions, (Arsenault) \$ 52
Alloy Strengthening Due to Atomic Order, (Marcinkowski) \$ 31

Massachusetts Institute of Technology

High Temperature Properties and Processes in
Ceramics, (Bowen, Wuensch) \$ 92
Thermal Neutron Scattering Studies of Molecular
Dynamics and Critical Phenomena in Fluids and
Solids, (Chen, Yip) \$ 80
The Luminescence Process in Chemical Reactions, (Gole) \$ 57

Massachusetts Institute of Technology, continued

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|---|-------|
| Basic Research in Crystalline and Noncrystalline Ceramic Systems, (Kingery, Coble) | \$408 |
| Low Temperature and Neutron Physics Studies, (Shull) | \$ 94 |

Michigan State University

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| Properties of Rare Gas Solids, (Pollack) | \$100 |
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Michigan Technological University

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| A Study of Grain Boundary Segregation Using the Auger Electron Emission Technique, (Stein, Heldt) | \$ 56 |
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University of Minnesota

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|---|-------|
| Analysis of the Ductile-Brittle Transition Temperature in Febinary Alloys, (Gerberich) | \$ 42 |
| Experimental Investigations in Solid State and Low Temperature Physics, (Goldman, Weyhmann, Zimmerman) | \$177 |
| In-Situ Electron Microscope Investigation of the Nucleation and Growth of Sputtered Thin Films (Hutchinson) | \$ 41 |

National Academy of Sciences/NRC - National Materials Advisory Board

| | |
|--|-------|
| Contingency Plans for Chromium Utilization | \$ 50 |
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National Academy of Sciences/NRC

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|--|-------|
| An Assessment of the National Need for Facilities Dedicated to the Production of Synchrotron Radiation, (Reed) | \$ 25 |
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State University of New York/Stony Brook

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| Applications of Microdynamics and Lattice Mechanics to Problems in Plastic Flow and Fracture, (Bilello) | \$ 54 |
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University of North Carolina

| | |
|---|-------|
| Investigation of Defect Structures by Electric Polarization and Relaxation Methods, (Crawford) | \$ 79 |
|---|-------|

North Carolina State University

Sorption of Cesium by Graphites at High
Temperatures, (Zumwalt) \$ 48

Northwestern University

Effect of Point Defects on Mechanical Properties
of Metals, (Meshii) \$ 54
Basic Research on Ceramic Materials for
Energy, (Whitmore) \$ 65

Ohio State University

Fundamental Studies of Metal Fluorination Reactions, (Rapp) ... \$ 65
Hydrogen Attack of Steel, (Shewmon) \$ 40
Corrosion, Stress Corrosion Cracking, and Electro-
chemistry of the Iron and Nickel Base Alloys in
in Caustic Environments, (Staehle, Agrawal) \$ 53

Oklahoma State University

Electronic Structure of Defects in Oxides, (Summers) \$ 23

Pennsylvania State University

Ceramic Research, (Bradt, Hoke) \$ 33
Studies of Mechanical Properties and Irradiation
Damage Nucleation of HTGR Graphites, (Thrower) \$ 30
Structure of Glasses Containing Transition
Metal Ions, (White) \$ 50

Purdue University

Transport and Thermodynamic Properties of Solids, (Grace) \$ 40
High Temperature Effects of Internal Gas Pressures
in Ceramics, (Solomon) \$ 52

Rensselaer Polytechnic Institute

The Effect of Welding Variables on the Solidification
Substructure, Mechanical Properties and Corrosion
Behavior of Austenitic Stainless Steel Weld Metal
(Savage, Duquette) \$ 43
Fatigue Behavior of BCC Metals, (Stoloff) \$ 30

University of Rochester

| | |
|---|-------|
| The Materials and Mechanics of Rate Effects in Brittle Fracture, (Burns) | \$ 43 |
| Diffusional Creep of Multicomponent Systems, (Li) | \$ 38 |

University of Southern California

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|---|-------|
| Electrical and Mechanical Properties of Oxide Ceramics, (Kroger) | \$ 55 |
| Grain Boundary Sliding During High-Temperature Creep, (Langdon) | \$ 70 |

Stanford Research Institute

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| Chemistry of Zirconium Related to the Behavior of Nuclear Reactor Fuel Cladding, (Cubicciotti) | \$135 |
|---|-------|

Stanford University

| | |
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| Photovoltaic Materials Research - II-VI Heterojunctions and Cu ₂ S/CdS Thin Films, (Bube) | \$ 80 |
| Structure Dependence of High Temperature Deformation of Metals, (Nix) | \$ 60 |
| Diffusion of oxygen in Liquid Metal Systems (Stevenson) | \$ 36 |

University of Tennessee

| | |
|---|-------|
| Microstructure-Property Relationships in Austenitic Stainless Steels, (Spruiell) | \$ 26 |
| Application of Adiabatic Calorimetry to Metal Systems, (Stansbury, Brooks) | \$ 37 |

U. S. Steel Corporation

| | |
|--|-------|
| Studies of Fundamental Factors Controlling Catalyzation of Reactions of Gases with Carbonaceous Solids, (Mahoney) . | \$ 75 |
|--|-------|

University of Utah

| | |
|---|-------|
| Positron Lifetime Measurements as a Non-Destructive Technique to Monitor Fatigue Damage, (Byrne, Ure) | \$ 49 |
| Impurity Effects on the Creep of Polycrystalline Magnesium and Aluminum Oxides at Elevated Temperatures, (Gordon) | \$ 36 |

Varian Associates

Research on Lattice Mismatched Semiconductor
Layers, (Bell, Antypas) \$ 70

University of Vermont

Thermodynamic and Transport Properties of Inter-
stitial Hydrogen Isotopes in Metal Systems, (Brown) \$ 22

University of Virginia

Electronic Properties of Metals and Alloys, and
Molecules, (Coleman) \$ 95

University of Washington

A Study of Phase Transformations and Super-
conductivity, (Polonis) \$ 11

University of Wisconsin

Void Nucleation and Growth in Heavy Ion and Electron
Bombarded Pure Metals, (Kulcinski) \$ 70

MATERIALS SCIENCES FY 1977 FUNDING
BY LABORATORY

Fig. 1

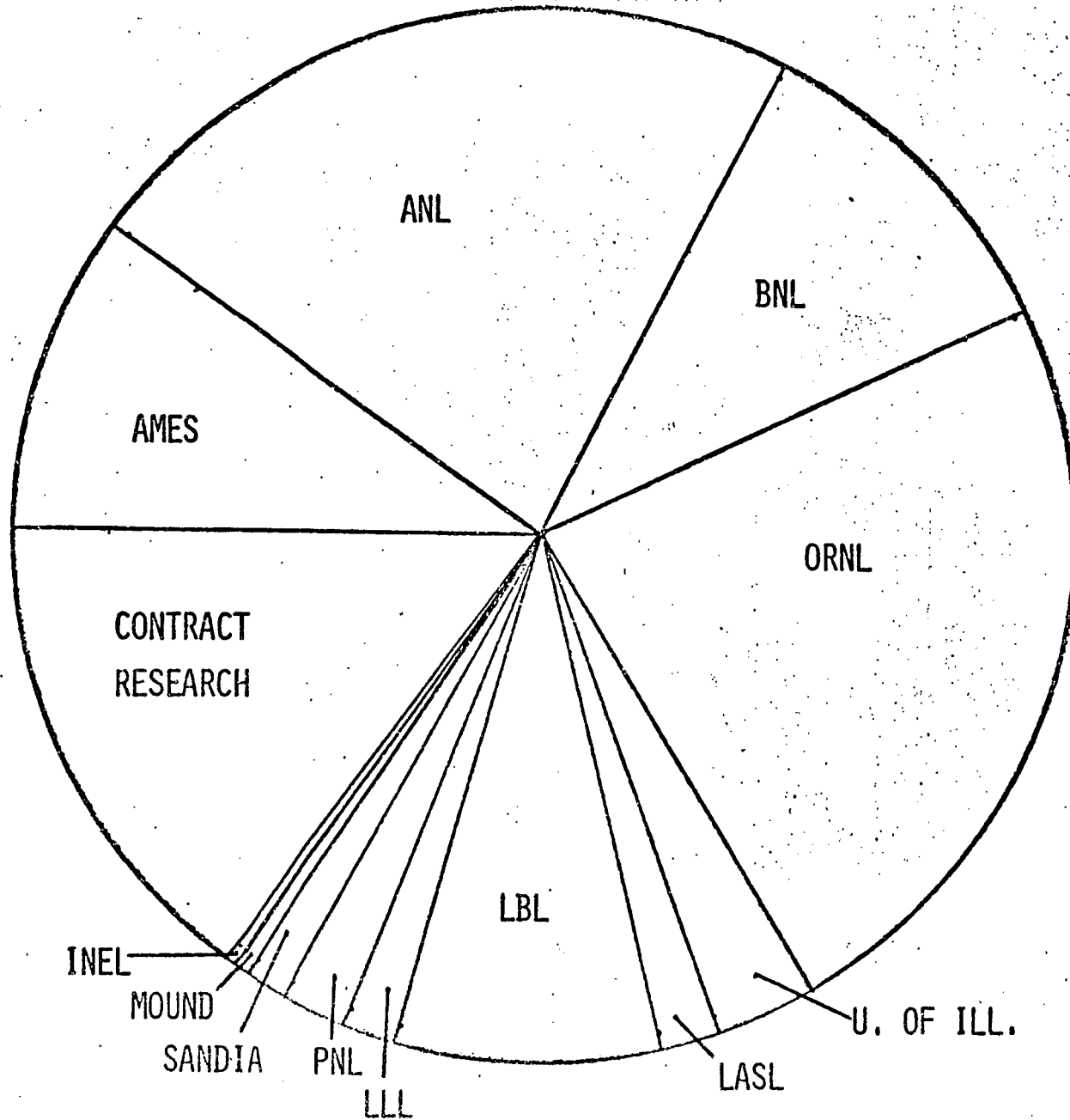
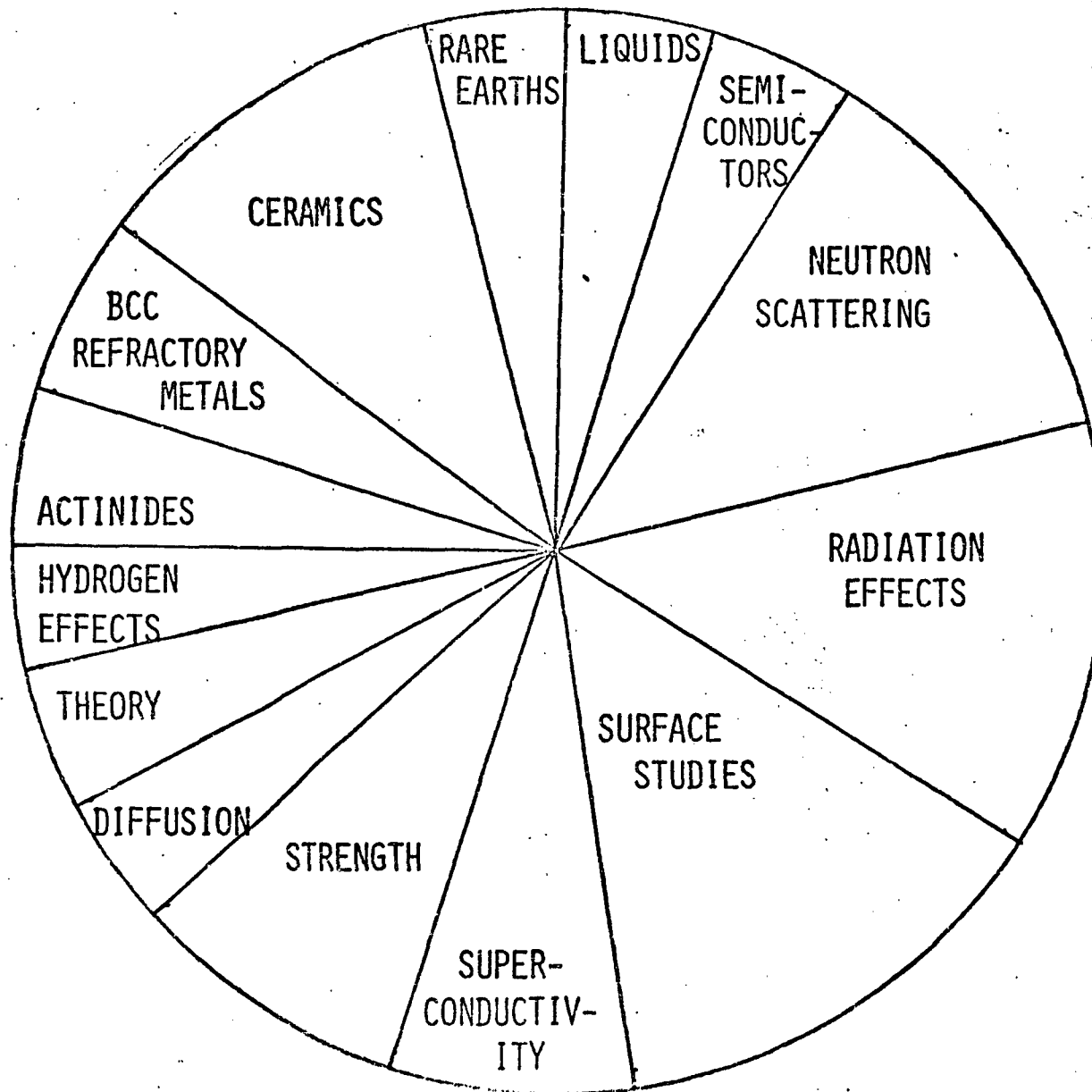


Fig. 2

MATERIALS SCIENCES FY 1977 FUNDING BY TOPICAL AREA



Title: Reference (U, Pu)O₂ LMFBR Fuel Development Program

| <u>Contract</u> | <u>Contractor</u> | <u>Principal Investigator</u> |
|-----------------|-------------------|--------------------------------|
| AL 0036 | LASL | G. R. Waterbury |
| CH 0038 | ANL | C. M. Walters |
| CH 3045 | WARD | A. Boltax |
| FF 2170 | HEDL | W. E. Roake |
| SF 893 | GE | E. A. Aitken and M. G. Adamson |

FY 1976 Funding Level
14247 K

Objective and Scope

To demonstrate safe and reliable performance of reference LMFBR fuel and blanket assemblies under steady-state and design transient conditions and establish the inherent performance capability, mode and consequences of fuel failure.

| | |
|---------|---|
| AL 0036 | Examination of unirradiated and irradiated LMFBR fuels and to develop the chemical analytical methods necessary to such examinations |
| CH 0038 | Gives direct support to LMFBR core-design efforts by providing experimental results and analyses on the in-reactor deformation and fracture of stainless steel |
| CH 3045 | Testing and evaluation of the critical features of mixed oxide fuel pin and radial blanket pin behavior in fast reactor environment |
| FF 2170 | Provides fuel/cladding data and physical/chemical behavior of mixed oxide/stainless steel clad fuel systems in a fast reactor environment |
| SF 893 | Understanding the chemical behavior of irradiated fast reactor mixed oxide fuels under normal and failed operation to provide a comprehensive basis for code prediction of fuel performance |

Title: Advanced LMFBR Reactor Fuel and Cladding Development

| <u>Contract</u> | <u>Contractor</u> | <u>Principal Investigators</u> |
|-----------------|-------------------|-------------------------------------|
| AL 0036 | LASL | J. L. Green and K. W. R. Johnson |
| CH 0038 | ANL | J. H. Kittel |
| CH 0092 | BMI | D. L. Keller |
| CH 2107 | Un. of Cinn. | J. Motteff |
| CH 3045 | WARD | M. L. Bleiberg and J. S. Theilacker |
| FF 2170 | HEDL | J. J. Laidler and J. E. Hanson |
| HQ 9999 | NRL | F. A. Smidt, Jr. |
| OR C022 | ORNL | V. J. Tennery |
| SF 0824 | AI | L. J. Jones |
| SF 894 | GE | W. K. Appleby |
| SF 0034 | UCLA | A. R. Wazzan |

FY 1976 Funding Level
13034 K

Objective and Scope

To develop advanced LMFBR fuel and core structural materials which are capable of producing breeding ratios of at least 1.30, doubling times of 15 years or less, and peak burnup capabilities of 150,000 MWD/T and introducing these systems into commercial fast reactors.

| | |
|---------|---|
| AL 0036 | Fabrication of mixed (U, Pu)C fuels for radiation testing in fast reactor environments, determination of such fuels stability under steady state and transient operation conditions, and to coordinate the national advanced fuels fabrication program. |
| CH 0038 | Evaluate the irradiation test results of the fast reactor advanced fuels program and to coordinate the advanced LMFBR fuel development program. |
| CH 0092 | Development of technology and fabrication processes for the production of mixed (U, Pu)N fast reactor fuels. |
| CH 2107 | Determine the effects of elevated temperature deformation on dislocation microstructure of LMFBR clad/duct alloy materials. |
| CH 3045 | Fabrication of mixed carbide fuel pins for testing in fast reactor environment; participation in the national program to develop advanced alloys for cladding and duct use. |

- FF 2170 Investigate a mixed oxide system which will meet the objectives of LMFBR fuels as stated above; central coordinator and major participant in the national program for advanced alloy development.
- HQ 9999 Participation in the national development for improved clad/duct alloy materials.
- OR 0022 Participation in the development and testing of advanced alloys for cladding and ducts.
- SF 0824 Design, fabricate, and test advanced mixed carbide fuels.
- SF 894 Participate in the national program for the design and testing of advanced core materials for LMFBR systems.
- SF 0034 Analyze and determine the performance of mixed carbide and nitride fuels for use in the steady state and non-equilibrium conditions in fast reactors.

Title: Materials Engineering

| <u>Contract</u> | <u>Contractor</u> | <u>Principal Investigator</u> |
|-----------------|-------------------|-------------------------------|
| CW 135 | WARD | G. R. Taylor |
| FF 088 | HEDL | H. H. Yoshikawa |
| IA 007 | ANC | D. W. Hood |
| IA 009 | ANC | G. E. Korth |
| OH 103 | ORNL | G. M. Slaughter |
| SA 015 | AI | R. V. Anderson |
| SG 029 | GE | C. N. Spalaris/P. Ring |

FY 1976 Funding Level

2042 K

Objective and Scope

To develop fabrication methods and manufacturing processes suitable for the economical production of LMFBR reactor plant components.

| | |
|--------|--|
| CW 135 | Determine effects of environment on cracking behavior and corrosion of piping. |
| FF 088 | Provide and maintain a materials properties data handbook. |
| IA 007 | Provide advancements in welding technology. |
| IA 009 | Develop mechanical properties data for Alloy 718. |
| OH 103 | Develop fabrication methods for large diameter pipe and fittings. |
| SA 015 | Develop flexible pipe joints. |
| SG 029 | Provide tubes and tubesheets, develop NDE application methods, optimize water chemistry and establish welding procedures for CRBRP steam generators. |

Title: Hardsurfacing Alloys

| <u>Contract</u> | <u>Contractor</u> | <u>Principal Investigator</u> |
|-----------------|-------------------|-------------------------------|
| CW 064 | WARD | P. Murray |
| FF 129 | HEDL | J. Spanner |
| SL 009 | LMEC | J. Droher |

FY 1976 Funding Level

820 K

Objective and Scope

This program involves development and testing of hardsurfacing techniques for use on mechanical interfaces which require low friction movement without self welding in a sodium or inert gas environment. Existing argument interface designs are worked up and tested for the performance of the hardsurfacing. Test data is reduced to design form and put in the Nuclear Systems Materials Handbook.

| | |
|--------|----------------------------------|
| CW 064 | Friction, Wear, and Self Welding |
| FF 129 | Hardfacing Alloy Development |
| SL 009 | Friction and Wear in Sodium |

Title: Sodium Technology

| <u>Contract</u> | <u>Contractor</u> | <u>Principal Investigator</u> |
|-----------------|-------------------|-------------------------------|
| SG 028 | GE | C. Spalaris |
| CA 024 | ANL | R. Weeks |
| CW 065 | WARD | P. Murray |
| FF 129 | HEDL | R. Cash |
| SA 007 | AI | J. Asquith |

FY 1976 Funding Level

2370 K

Objective and Scope

This program is delineating the effects of a sodium environment on LMFBR structural alloys for fuel cladding, piping, vessels, steam generators and an elastomeric seal material. The scope includes corrosion, mass transfer and mechanical property studies. The data is reduced to design data form and then put in the Nuclear Systems Materials Handbook. Work involves existing commercial alloys and advanced, developmental alloys and elastomers.

Title: Structural Alloys - Fabrication

| <u>Contract</u> | <u>Contractor</u> | <u>Principal Investigator</u> |
|-----------------|-------------------|-------------------------------|
| OH 028 | ORNL | P. Patriarca |

FY 1976 Funding Level

1150 K.

Objective and Scope

The purpose of steam generator materials development is to provide engineering design data and information on materials necessary for fabrication and operation of steam generators for CRBRP, PLBR, and CBR-1 plants. This includes development for both the IHTS and sodium-water steam generators.

Title: Structural Materials -- Mechanical Properties

| <u>Contract</u> | <u>Contractor</u> | <u>Principal Investigator</u> |
|-----------------|-------------------|-------------------------------|
| CA 008 | ANL | D. Diercks |
| FF 125 | HEDL | R. Knecht |
| HH 010 | NRL | L. Steele |
| OH 024 | ORNL | G. M. Slaughter |
| OH 038 | ORNL | P. Patriarca |
| OH 050 | ORNL | C. Brinkman |
| SC 007 | UCLA | A. Tetelman |

FY 1976 Funding Level

2443 K

Objective and Scope

The purpose of the structural materials mechanical property programs are to provide irradiated and unirradiated data and structural analysis for the design, operations, support and safety analysis of FTR/CRBRP/FLBR components and structures. This includes development to obtain design data, etc., on base materials and weldments for commercial and developmental structural alloys.

| | |
|--------|--|
| CA 008 | Fracture and Low Cycle Fatigue |
| FF 125 | Radiation Effects on Structural Materials |
| HH 010 | Advanced Structural Materials Development |
| OH 024 | Mechanical and Metallurgical Behavior of Weldments for LMFBR |
| OH 050 | Mechanical Properties for Structural Materials |
| SX 007 | High Temperature Deformation and Fracture of Reactor Steels |

Title: NDT

| <u>Contract</u> | <u>Contractor</u> | <u>Principal Investigator</u> |
|-----------------|-------------------|-------------------------------|
| CA 009 | ANL | K. Reiman |
| FF 029 | HEDL | J. Spanner |
| OH 061 | ORNL | R. McClung |

FY 1976 Funding Level

435 K

Objective and Scope

The purpose of the development effort is to establish nondestructive test techniques, standards, procedures and equipment to assess the quality and integrity of materials in fabricated components and reactor systems. The scope includes development for pre-service and in-service inspection (ISI) methods for LMFBR reactor systems and components.

| | |
|--------|--|
| CA 009 | NDT Support-Components, Assemblies, Pins |
| FF 029 | Nondestructive Application Engineering |
| OH 061 | Nondestructive Testing |

DMA MATERIALS RESEARCH AND DEVELOPMENT PROGRAM

The DMA Materials Research and Development Program is directed toward basic material science, the understanding and development of advanced materials and fabrication technology, and the development of material and processes required to produce nuclear and nonnuclear parts. These materials research and development programs are contained in our budget under the following categories.

Development Capability

- Supporting Research: Includes material research which is fundamental to weapon development. Work is directed toward development of analytical procedures and nondestructive test methods needed to support ongoing weapon development programs and toward development and characterization of materials. Supporting materials research is required where the design engineer's ability to apply materials is limited by lack of understanding of basic materials phenomena. Typical research areas include: plutonium and actinide research, chemical high explosives, inorganic-material synthesis, chemical-characterization support, material-characterization studies, surface studies, theoretical material research, equation of state, nondestructive testing, structural materials and electronic materials. This category of research is performed at the three weapons laboratories--LASL, LLL, and Sandia. The total estimated funding for FY 1976 is \$9.6M.
- Materials and Fabrication Technology: Includes the understanding of advanced materials and their potential application through studies of the mechanisms which influence material behavior, failure criteria, determination of properties, and the development of processes to formulate new materials. Specifically includes the application of materials to engineering techniques and the development of fabrication technology. This materials program is directed toward providing well characterized materials for long-term predictable weapon concepts. This research is performed at the three weapons laboratories--LASL, LLL, and Sandia. The total estimated funding for FY 1976 is \$16.4M.

Production and Surveillance

- Process Development: Includes work done by the production plants in the development of material and processes required to produce nuclear and nonnuclear parts. Development work is performed on new materials and production processes for weapon concepts in advanced development, engineering development, and production phases. It also includes manufacturing research and development work to improve existing processes for efficiency and safety. This program is conducted by the seven production contractors--Bendix, Rockwell International, Monsanto, General Electric, Mason and Hanger, Union Carbide and duPont. The total estimated funding for FY 1976 is \$33M.

The following pages present selected material program summaries of the three weapons laboratories. These summaries are not presented in the budget category formats discussed above. The funding levels are estimates only. In addition, these summaries do not include all the material research being conducted by the laboratories. Summaries have not been prepared by the production contractors since most of the efforts are classified.

LASL Materials Program

METALLURGYPlutonium Metallurgy

Principal Investigator: R. N. R. Mulford
FY 1976 Funding: \$123K

A vigorous effort exists to determine the mechanical properties of pure plutonium and plutonium alloys. The conventional properties such as uniaxial tensile and compressive strengths and elongations are measured over a range of strain rates from 10^{-5} to 100 per second. The influence of processing heat treatments and fabrication variables on the mixture and stability of plutonium allotropic phases and the resulting mechanical properties is investigated. Future plans involve the investigation of the effects of multi-axial stress on these alloys. Stresses encountered in actual applications are almost always multi-axial, and mechanical properties of a metal are frequently a strong function of stress biaxiality. It is thus important to develop data on material properties that are more detailed than the conventional uniaxial tensile measurements. Such information does not now exist for plutonium and its alloys.

Phase Relations in Plutonium Metallic Systems

Principal Investigator: R. N. R. Mulford
FY 1976 Funding: \$300K

This project has in the past produced a considerable body of knowledge about binary and ternary composition-temperature phase diagrams. The quantity of effort in this category has diminished recently, mainly because most of the metallic elements in the Periodic Table have been covered. However, there is currently some effort devoted to determination of the structures of inter-metallic compounds, particularly the compounds of plutonium with the noble metals Pt, Ir, Os, and Pd. In addition, phase relations of a few special plutonium systems containing low concentrations of alloying element are being investigated with special attention to transition temperatures, impurity effects, rates of transition, and the structures of the phases resulting from transitions. In connection with binary plutonium phase systems, we are doing splat-cooling experiments in which alloys are quenched very rapidly from the liquid state by firing a droplet of liquid onto a cooled target. Non-equilibrium phases are produced. Knowledge of the properties of the nonequilibrium phases improves our understanding of the properties of equilibrium phases and contributes to understanding of the bonding in plutonium alloys.

We have also done high pressure phase studies on plutonium and its alloys. Equipment is available which will reach 60 kb under static conditions. P-V-T diagrams have been produced which define the ranges of stability of the various plutonium phases.

A small effort exists to develop better techniques for bonding various metals to plutonium.

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Computer Simulation of Metal Casting

Principal Investigator: D. J. Sandstrom
FY 1976 Funding: \$52K

A continuing study is under way to develop computer codes and graphics techniques for modeling metal casting processes. This technique allows one to design casting molds to optimize mold design and subsequent properties and casting quality. We are currently using standard heat transfer codes to study the mold filling and solidification process for uranium metal castings.

Micro-Computer Control of Electron Beam Welding Equipment

Principal Investigator: D. J. Sandstrom
FY 1976 Funding: \$70K

We are currently performing a feasibility study aimed at demonstrating the practicality of using microprocessors (micro computers) for controlling a very complex electron-beam welding machine. This type of control will allow us to operate electron-beam welding equipment with highly reproducible parameters at levels of beam voltage and current not heretofore deemed possible.

Fabricability and Weldability of High Strength Austenitic Stainless Steels

Principal Investigator: D. J. Sandstrom
FY 1976 Funding: \$70K

The LASL in cooperation with other ERDA contractors has been involved in an in-depth study on the weldability and fabrication of high strength austenitic stainless steel. The stable austenitic stainless steels such as Armco Steels 21-6-9 alloy have mechanical properties which make them particularly attractive for use at cryogenic temperatures and in the presence of H_2 and its isotopes. This class of materials has exhibited erratic welding properties and in order to gain utilization of the desirable properties of these alloys we must solve the welding problem.

Development of High Density, High Z Structural Materials

Principal Investigator: D. J. Sandstrom
FY 1976 Funding: \$139K

A continuing effort aimed at the development of high density, high Z number, nonfissile materials is planned. Materials of interest include plasma-arc shaped W which can be subsequently infiltrated with lower Z, lower density materials for purposes of strengthening. Additional efforts are under way in the area of fabricating reproducible alloy material based upon the power metallurgy processing of W-Ni-Fe and W-Ni-Cu alloys.

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Thermal Diffusivity of Pu and Its Alloys

Principal Investigator: W. J. Maraman
FY 1976 Funding: \$71K

Our thermal diffusivity studies have resulted in a reliable, easily applied technique for making measurements on a variety of Pu and Pu alloy specimens by employing a flash laser heat source. By using well characterized Pu alloy specimens and "crack-free" Pu specimens in this program we are now able to generate meaningful, reproducible thermal diffusivity data up to the melting point of Pu.

Applied Metallurgical Studies on Pu and Its Alloys

Principal Investigator: W. J. Maraman
FY 1976 Funding: \$781K

Our applied metallurgy studies have resulted in the development of several new alloys. We have maintained capabilities for the preparation of Pu components and specimens. We are continuing the development and complete characterization of new alloys made by cast, wrought and powder metallurgical techniques.

Materials Characterization

Principal Investigator: R. D. Reiswig
FY 1976 Funding: \$113K

The techniques of optical microscopy (metallography), electron microscopy (both transmission and scanning), and x-ray diffraction phase identification are used to characterize a wide variety of materials resulting from the Laboratory's materials research programs. The materials include metals, alloys, ceramics, solid compounds, cermets, and graphite-base composites. Such characterization is key to the progress of materials research.

Electroforming of Various Metals and Alloys

Principal Investigator: D. J. Sandstrom
FY 1976 Funding: \$87K

A continuing study is under way toward perfecting processes of electroforming a variety of metals for structural applications. Electroforming techniques for manufacturing pressure vessels out of a variety of materials which can be electro-deposited from aqueous solution have been developed. Other structural components and assemblies requiring unique corrosion resistance have been developed.

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CHEMISTRYChemical Characterizations of Materials

Principal Investigator: Glenn R. Waterbury
FY 1976 Funding: \$677K

Chemical analyses will be continued to characterize a multitude of metals, alloys, compounds, ceramics, cermets, and mixtures. This support operation will be augmented by research and development of new analysis capabilities to provide the needed methodology for extensive characterizations of specific materials. The analytical capabilities, which now include most analysis techniques with the recent addition of ion microprobe, energy-dispersive x-ray fluorescence, automated mass spectrometry, automated trace oxygen measurements, and new atomic absorption applications, will be expanded to include the new surface analysis techniques of electron and Auger spectroscopy, rapid direct reading emission spectroscopy, and automation of other methods.

Lithium Hydride, Deuteride and Tritide Studies

Principal Investigator: J. D. Farr
FY 1976 Funding: \$144K

We shall continue our studies of the synthesis and properties of lithium hydride using primarily the heavier isotopes of hydrogen. The two general areas of interest are (1) the preparation and characterization of LiT samples for our ^3He release studies and (2) the exchange reaction between LiD and T_2 gas. These two areas have in common the need to be able to compare the surface areas of different samples. We will use the BET technique as a means of comparison. An in-line calorimeter should be installed in our tritium drybox line for measuring the T content of samples. Along with this work we are also looking at ways to continually reduce the tritium release from our facility.

A System for Studying the Initial Stages of Gases Reacting with Metals

Principal Investigator: J. D. Farr
FY 1976 Funding: \$79K

An ultrahigh vacuum system was constructed for use in conjunction with a Rodder ultramicrobalance for studies of uranium corrosion. The use of sorption, titanium sublimation and Noble ion pumps avoids the need for oil-diffusion pumping thus eliminating the possibility of sample contamination by oil vapor. Provisions for in situ sputter cleaning of samples as well as electron beam heating and infrared thermometry have been built into the system.

It is proposed to carry out a systematic investigation of the oxidation of high purity uranium from 25 to 300°C . The physical and chemical properties of the oxides formed will be studied by diffraction, microscopy and kinetic techniques. In particular, the reactivity of the partially oxidized coupons in H_2 atmospheres will be related to the surface preparation, composition and oxidation treatments.

Superconductivity Studies

Principal Investigator: J. D. Farr
FY 1976 Funding: \$216K

The effort in the field of superconductivity is being pursued in two general areas: (1) the development of a technique for the production of long lengths of electrical conductors having a continuous superconducting layer of high T_c material and (2) studies on well characterized known high T_c compounds and on new binary and ternary compositions. Considerable success has already been achieved in the development of a chemical vapor deposition (CVD) process which produces continuous and adherent coatings of Nb_3Ge material on copper tubing over a 40 cm length. The material has a T_c of 22 K and a critical current density of 1.8×10^6 A/cm² at 13.8 K. This work is being continued and expanded to include the design and construction of a continuous CVD coating unit which will permit the preparation of a continuous coating on multimeter lengths of copper pipe. The studies on known high T_c materials (e.g., Nb_3Ge , Nb_3Ga , $Nb_3(Al,Ge)$, and NbN) as well as on new compositions (e.g., Mo-Re, Mo-Tc, W-Re, W-Tc) will involve preparation by the usual high temperature methods as well as by two "low temperature" methods (sputtering and CVD). These studies are presently under way and will be continued. A small experimental sputtering unit and a small CVD apparatus have been constructed which permit the preparation of small samples of materials on heated substrates at controlled temperatures. The objective of these studies is the correlation of composition and structure with superconducting properties for these materials. A comparison of the superconducting properties of similar compositions prepared by the different methods should also prove of great interest.

Release of 3He from Solid Tritides

Principal Investigator: J. D. Farr
FY 1976 Funding: \$22K

A program to study the release of 3He from LiT stored under an over-pressure of T_2 was started. The samples, containing equal amounts of tritium in the gas and solid phases and stored at room temperature, are being monitored by periodically measuring the quantity of thermal neutrons absorbed by 3He in each phase. A neutron diffractometer was modified to permit measuring the attenuation, by the sample, of a monochromatic thermal neutron beam.

Process Development for Polymeric Materials

Principal Investigator: D. J. Sandstrom
FY 1976 Funding: \$87K

A variety of polymeric materials are required in all phases of energy, safeguards, and defense related work. A continuing program aimed at the development of improved manufacturing processes and materials will be conducted.

Surface Studies

Principal Investigator: W. P. Ellis
FY 1976 Funding: \$85K

The most modern techniques, particularly low energy electron diffraction (LEED), inelastic secondary electron spectral analyses (Auger and loss spectra), and photoelectron spectroscopy (PES), are used to characterize surface structures, to detect and identify surface impurities, to obtain information about valence band electrons, and to study gas-solid reactions from their inception. An ultra-high-vacuum PES system, that is the most advanced in the world for studying surfaces and reactions of reactive metals such as uranium and plutonium, is being put into use. This system will be used to study hydriding and passivation of these metals. Work will be done on heterogeneous catalysis. Fundamental studies using the above electron probes will be correlated with practical studies of the catalytic activity of powders using a flow reactor system with chromatographic analysis of product gas mixtures. The catalytic activity of metals and of compounds of uranium, rare earths and other elements will be studied. The surface studies are relevant to ERDA programs on synthetic fuel production. These include both older processes such as coal gasification and new processes such as thermochemical hydrogen production.

Plutonium Electrochemistry

Principal Investigator: W. J. Maraman
FY 1976 Funding: \$213K

Our electrowinning studies have resulted in reliable processes for producing high purity plutonium and neptunium on a useful scale from impure metals and alloys. We have undertaken the development of an economical method for electrowinning plutonium directly from its oxide. We are continuing the determination of thermodynamic properties of plutonium, its alloys and compounds by measurements of EMF cells above 650°C.

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PHYSICSActinide Metal Physics

Principal Investigator: R. N. R. Mulford
FY 1976 Funding: \$219K

This activity is directed towards the achievement of a basic understanding of the electronic and physical properties of the actinide metals (especially plutonium), their alloys and intermetallic compounds. Closely interacting experimental and theoretical studies are carried out on the superconductivity, magnetism, and general electronic features of various actinide systems. Attention is concentrated on the mechanisms for bonding extant in these systems, especially as concerns the participation of 5f electrons.

Rather than using band theoretical studies to detail such exotic features of metals as, e.g., their Fermi surface topologies, the calculations and accompanying experiments are being aimed at elucidating the essential bonding phenomena appropriate to materials problems. These efforts are proving informative for materials behavior throughout the periodic table, and, somewhat ironically, the theoretical foundation of the approach used could only have been discovered by considering the relatively complex actinide metals--this because of our new discovery of the chemical bonding behavior of 5f electrons in metallic systems.

LLL Materials Program

METALLURGYBeryllium

Principal Investigator: J. Hanafee
FY 1976 Funding: \$100K

Significant improvements have been realized in properties such as yield strength, ultimate strength, and ductility through the development of both improved chemistry control and new manufacturing methods. Recent work on beryllium consists of: Prediction of fracture toughness of thin sheet material by the use of J integral methods and study of the effect of yield strength, grain size, impurity level, and microstructure on the flow and fracture behavior of materials produced by new processes.

Plutonium and Plutonium Alloys

Principal Investigator: J. Hauber
FY 1976 Funding: \$750K

Developmental work on plutonium and its alloys involves study of: Phase diagrams; phase transformation and its influence on parameters such as density; effect of impurities on the response of the metal; and the determination of basic structural and physical properties.

Fabrication and development procedures which are routinely performed on most materials require special handling facilities and expertise when they are performed on plutonium. For this reason, Metallurgy Division has developed capabilities for casting, welding, brazing, rolling, mechanical testing, vapor coating, metallography and chemical analysis.

Ferrous Metals

Principal Investigators: P. Landon & R. Vandervoort
FY 1976 Funding: \$100K

Basic studies being conducted entail determination of the influence of impurities on the weldability of 21-6-9 stainless; study of low temperature reversion, stacking fault energy and the relationship of these variables to the susceptibility of stainless steels to hydrogen embrittlement; correlation of properties and metallographic structure with thermal-mechanical history.

Metallographic Analysis

Principal Investigator: J. Johnson

Included here are optical metallography, microprobe analysis, scanning and transmission microscopy, x-ray, as well as fractographic characterization. These activities support all aspects of the metallurgy program, but they are particularly important to the success of the materials and process development endeavors.

Mechanical Properties

Principal Investigator: R. Vandervoort
FY 1976 Funding: \$100K

Determination and Evaluation: Basic considerations involve determination of the effect of variables such as stress, temperature and strain rate on the mechanical response of metals. Tests commonly run are creep, fatigue, fracture toughness, tensile, compressive and torsion. Also tested are properties such as weld and bond strength.

Physical Properties Determination and Evaluation: Another aspect of the characterization endeavor is the determination of physical properties of metals. Metallurgy capabilities in this area are primarily devoted to precision density determination, phase identification, drop calorimetry for enthalpy measurement, and DTA studies of melting points and phase transformation.

Corrosion Effects: Two test units are available for evaluation of the susceptibility of materials to embrittlement in a high pressure hydrogen environment. Tensile, fracture, and static load delayed failure tests can be conducted in hydrogen gas up to pressures of 69 MPa. Test temperatures can be varied from 23-200°C, and strain rate can be varied from 10^{-5} to 10^{-2} /s.

Chemical Vapor Deposition

Principal Investigator: W. R. Holman
FY 1976 Funding: \$300K

Fabrication processes are being developed using chemical vapor deposition as a means of producing special shapes. This activity has included an indepth study of the fundamental mechanisms involved in the deposition process as well as a great deal of iterative design work to produce highly sophisticated equipment.

Physical Vapor Deposition

Principal Investigator: W. R. Holman
FY 1976 Funding: \$50K

High-rate thermal evaporation of metals from electron beam heated sources is used to promote improved physical or chemical properties of metallic or nonmetallic surfaces. Examples are bonding and diffusion aids, protection (diffusion and corrosion barriers), thermal and electrical conductivity (plastics).

Electroforming

Principal Investigator: H. Wiesner
FY 1976 Funding: \$50K

Continuing study of developing processes of electroforming a variety of metals for structural applications.

LLL Materials Program

ORGANIC MATERIALSPolymeric Materials Technology

Polymer chemistry has advanced from an art to a highly theoretical science in a relatively few decades. Understanding the reaction kinetics, the influence of molecular weight, degree of crosslinking, branching, crystallinity, and dipole groups in the polymer chain, is prerequisite to understanding, predicting, and tailoring polymers to requirements. Recent developments such as stereospecific polymerization have shown that even the conventional monomers can be made into improved polymers. Non-linear polymerization and copolymerization reaction have the potential for producing high performance adhesives, elastomers, and fibers. The Kevlar 49 fiber is an excellent example of what can be accomplished with a theoretically designed polymerization effort. With the cooperation of physicists, physical chemists, polymer chemists, and engineers, new materials are being designed and older ones modified to satisfy exacting requirements.

Adhesives

Principal Investigator: H. George Hammon
FY 1976 Funding: \$300K

- Adhesive Synthesis: The current program is to synthesize urethane prepolymers for adhesive and potting application. The approach is to synthesize MDI and Hylene W polyether prepolymers and to cure these with polyols and amines.

- Variable Modulus Room Temperature Adhesive: The goal of this formulation program is to provide a general purpose adhesive that can in part be tailored by the user. The user should be able to make trade-offs between working time, viscosity, and modulus by varying the proportions of the system. The initial approach is to combine epoxy and urethane prepolymers.

- High Explosive Adhesives: This task is to formulate an adhesive for TATB. The approach is to evaluate standard adhesives with each of the three candidate binders for the TATB, Viton A, Estane, and Kel-F.

Polymer Formulation Program

Principal Investigator: L. E. Peck
FY 1976 Funding: \$150K

This program exploits the entire array of commercially available materials for various applications. Formulating is done to obtain optimum product performance, processing characteristics, or special properties required. Current formulating tasks include:

- Adhesive Formulations: Optimize the three adhesive and potting formulations; (1) HGH-2, Adiprene L-315/Polyol/FeAcAc, (2) HGH-3, Adiprene L-315/Polyol/DABCO, and (3) HGH-4, Adiprene LW-520/TONOX, with respect to processing, cure behavior, aging characteristics, and compatibility. This is the stage where the adhesive application and performance aspects dominate development.

Consideration of working time, effective bond thickness, and bond strengths are investigated. Surface preparation and substrate characteristics all have to be evaluated. The synthesis program will phase into this stage soon.

- 10B Formulation: Develop polymer formulations that will accept high particulate filler loadings and be functional at temperatures to 900°C for short periods of time. The approach is to select multi-modal particle size blends and elastomeric matrices such as VCE. Particle characterization lab supports this effort.

- Matrix Formulating: Formulate high performance epoxy polymers for adhesive, laminating and fiber composites. The approach is to control the stoichiometry and degree of cure of a few selected systems and correlate these with the processing and thermal-mechanical properties.

- Cushion: An earlier polymer synthesis program developed an improved silicone polymer. The current polymer is manufactured by a single supplier exclusively for the AEC under an agreement which expires in June 1975. The goal of this task is to use the more available polymer which has improved processing characteristics and mechanical behavior (less compression set). The cellular product is formed with urea as a temporary filler. Urea is in short supply and interacts with the silicone curing system. A second goal of this task is to certify an alternate temporary filler.

Polymer Process Development Program

Principal Investigator: J. K. Lepper
FY 1976 Funding: \$150K

The performance of polymeric materials is highly dependent upon their processing history. In addition, an integrated polymer program needs roots; if you can't process the material and make a product it wasn't worth developing. Current activities include developing methods for processing high temperature polymers for structural and electrical laminates. Included in this task is the development of new processing equipment such as the computer controlled filament winding machine and the programmable high temperature laminating press.

Process development capabilities must be maintained at design laboratories in order to transfer advance technology to production agencies and value production problems.

High Explosives Technology

The organic Materials Division is responsible for development of high explosives. Explosives used may be: A PBX, such as LX-04, LX-10, or LX-14; an ECS such as RX-08-A3; an extrudable such as LX-13; an improved acceptor HE; a paste HE; a heat powder, or a detonator or MDF material. All of these materials have different properties and different requirements, which must be measured, understood and controlled.

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The activities necessary to provide these materials can be categorized as follows:

Study of Chemical Physics

Principal Investigator: E. Lee
FY 1976 Funding: \$400K

An effort in chemical physics is by its nature, basic and general in scope. The specific examples are studies not aimed at specific weapon or design concepts, but are aimed toward improving our knowledge and techniques. In order to select an HE with the specific properties needed to meet the design requirements of a given device and to properly control those properties it is essential to understand the chemical physics involved. An understanding of this sort of basic behavior is also needed to predict how a material will respond under normal/abnormal conditions. Some of the phenomena involved are:

- Detonation Physics: The relation of thermodynamics, hydrodynamics, and rapid chemical energy release mechanisms is the key element in detonation physics. Better understanding of detonation theory is needed to predict explosive performance and to allow the design of new HE's with enhanced attributes. Detonation Physics includes the concept and execution of experiments and the development of physical models and techniques for numerical analyses. The products of detonation physics research are equations of state and hydrodynamic descriptions or models.

The use of large-scale computer codes is vital to the activities included under detonation research. Several codes provide an analytical description of hydrodynamic test results. This allows the interpretation of experiments in terms of a model for explosives and metal behavior during metal acceleration. Another code is currently used to predict detonation parameters. These data and the results of hydrodynamic experiments are used to formulate an EOS for an explosive. HE equations of state have been developed for all main charge explosives and all auxiliary explosives used at LLL. Approximately 50 explosive EOS's have been published. Computer codes may also be used to predict properties of significant new materials and new formulations to guide the synthesis and formulation effort.

- Kinetics: This involves understanding the rate at which chemical reactions take place and is important in understanding both the very short-term (detonation) behavior and the long-term (aging) behavior of materials. It is also needed in understanding behavior under abnormal thermal conditions such as an accident or fire. Kinetics includes designing and executing experiments and developing physical models and numerical analysis techniques. The end result of research in kinetics is the ability to predict thermal stability and aging phenomenon and predictions of behavior under accident and fire conditions.

- Initiation: This involves understanding how the reactions that produce a detonation start, buildup, and propagate. This understanding is needed both for predicting and improving safety and for providing reliability in initiation systems. The study of initiation includes designing and executing experiments and developing physical models and numerical analysis techniques.

- Mechanical Response: This involves understanding the mechanical behavior of HE under a wide range of conditions from creep to shock. This knowledge is needed for improving the mechanical properties of HE's and in understanding initiation mechanisms. There exists a close interaction of HE mechanical response and safety (accidental initiation). Study of mechanical response includes designing and executing experiments and developing models and analysis techniques.

Materials Development

Principal Investigator: M. Finger
FY 1976 Funding: \$200K

Materials development is vital to provide new explosives. The major elements of a materials development program are:

- Syntheses*: No material program can exist for long without investigating new materials. These must be supplied by synthesis either internal or external to the Lab. Even where the actual program is carried out external to the Lab, an in-house capability is required for monitoring and guiding research. For the HE area expertise is required in aromatic nitration chemistry, aliphatic nitration chemistry, fluorocarbon chemistry, the chemistry of heterocyclic nitro-compounds, and polymer chemistry. The end product of this effort is the input for new HE formulation development described below. Currently there is no internally supported synthesis program.

- Formulations: Newly developed input materials must be formulated into useful end products with properties tailored to specific design requirements. Properties that must be considered include: density, mechanical strength-- in both tension and compression over a wide temperature range, bondability, chemical compatibility with other materials of construction, thermal properties, initiability, handling safety, energy output and energy output rate, etc. These properties are controlled by controlling formulation parameters such as temperature, pressure, composition, particle size and particle size distribution, contact times, agitation rates, shear rates, order and rate of addition of ingredients, use of surfactants, nature of the solvents, etc. The end product of this effort is a molding power suitable for fabrication or a material such as a paste HE ready to load into a test device.

- Fabrication: A new formulation in most cases must be fabricated into useful form. This normally involves mechanical or isostatic pressing, cutting, machining, drilling, and sometimes bonding. Pressing parameters are controlled to produce specified densities (usually the maximum obtainable) and to optimize mechanical properties. Results are fed back into the formulation effort and to the manufacturing facilities to guide them in further development. To achieve uniform high density and thereby obtain high energy, an iteration of pressing techniques and formulation variables such as particle size is often required.

*Unsupported Activity

- Process Development: After a new material is developed, its processing must be scaled up to production sized equipment. It is often not possible to reproduce exactly the conditions of the development equipment, and, therefore, modifications to the procedures are sometimes required. Most of this work must be carried on at the production agencies, but liaison and coordination is required from OMD. In order to guide and advise production agencies we must maintain our technical credibility by getting our hands "dirty"; that is, keeping skilled in this area by performing work.

Characterization

Principal Investigator: M. Finger
FY 1976 Funding: \$250K

A vigorous characterization program spanning a broad spectrum of disciplines is essential to provide guidance to materials development, to develop measurement techniques for use in the study of chemical physics and to provide input data on new materials. Areas that must be investigated include:

- Physical Characterization: This information for the most part is required in the selection of input materials for the formulation effort. The required measurements include: Density, particle size and particle size distribution, molecular weight and molecular weight distribution of binders, crystallography, and viscosity.

For each of these techniques we must develop measurement methods, develop data reduction and correlation methods and maintain equipment and operator skills as well as make the individual measurements required by various users.

- Mechanical Characterization: Mechanical characterization needed for the design of devices includes: Tensile strength as a function of temperature and strain rate, compressive strength as a function of temperature and strain rate, hugoniot data, and shear modulus.

- Chemical Characterization: Chemical characterization is needed to guide the formulation effort in selection of processing conditions. It is also needed as guidance for the compatibility and polymer programs, in selecting adhesives for bonding and in selecting cross-linking and surface active agents. Some of these activities are provided by the General Chemistry Division. Chemical characterization involves: Identification of functional groups such as nitro, determination of reactivity with other materials of construction, determination of decomposition rates, and toxicity.

- Thermal Characterization: Thermal properties must be known as input data for chemical physics studies, particularly kinetics, but also for thermochemical and thermodynamic information as guidance for materials development, and as input to the design teams. Certification of mock materials to insure the safety of experiments involving mock HE and fissile materials is also provided. Thermal properties of interest include: Melting points, boiling points, vapor pressures, thermal conductivity*, heat capacity**, time to explosion**, enthalpies*, combustion calorimetry*, and thermodynamic function determinations*.

*Not Supported

**Partially Supported

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- Initiation Characterization: This information is required to insure reliable lighting from the initiation train. Measurements include: Gap tests, wedge tests, gas gun experiments, aquarium tests and HE driven-flyer plate experiments.

All HE's are characterized by the level of energy required to cause reaction, the time and distance to build to steady state detonation; and the response of the HE at levels just below reaction. Unreacted Hugoniot and time to explosion are important data for response codes and design of initiation systems.

- Performance Characterization: These measurements are required as input to the detonation physics studies and as input to the design codes chiefly in the form of equations of state. Measurements used include: Cylinder tests, sphere tests, detonation calorimetry, magnetic particle probe, and free surface velocity.

- Safety Characterization: Safety testing includes: Drop weight impact test, susan test, skid test, vertical drop test, Site 300 drop tower test, and friction test.

Questions of safety will always be of prime importance in the field of explosives. The tests above yield results which are used as a basis for judging what kind of stimulus an explosive can be safely exposed to without undue risk of initiation.

LLL Materials Program

GENERAL CHEMISTRY ANALYTICAL CAPABILITIES AND SERVICES

Principal Investigator: K. Ernst
FY 1976 Funding: \$500K

Basic Applications

A low level of effort is applied to long range research aimed at ultimately providing better measurement techniques. The long range goals are to either provide new capabilities, faster information turn around time; greater sensitivity or greater precision or accuracy. Part of this effort is directed toward computer automation of chemical measurement systems, experimental design and information retrieval and data processing. Research is going on in the fields of microwave spectroscopy, chemiluminescence, and surface analysis.

Development of Analytical Techniques

New analytical methods are either adapted from published literature sources or are developed by our staff of physical, analytical, theoretical chemists and physicists.

We continually attempt to upgrade and improve our services.

Analytical Services

Based on program requirements, analytical services are provided in the following categories: (1) gas analysis, (2) elemental analysis, (3) surface analysis, (4) functional group analysis, (5) explosive analysis, and (6) other properties and measurements.

Compatibility

Principal Investigator: H. Leider
FY 1976 Funding: \$1150K

The Compatibility Group uses an integrated experimental and modeling approach which (often) yields understanding and a quantitative description of the synergistic interactions among materials as encountered in special environments.

LLL Materials Program

INORGANIC MATERIALS FABRICATION DEVELOPMENTFabrication Development

Principal Investigator: R. Landingham
FY 1976 Funding: \$550K

Cold Press and Sinter: This approach to fabricating high density ceramics is the most conventional method used today. The Fabrication Technology Group has the capability of applying this technology to nonconventional materials that are both radioactive and pyrophoric. We are somewhat unique in this regard because all processing operations from batching to the machining or grinding of the final product can be handled in box line systems with high purity argon. In addition to being able to handle such special materials, we have equivalent facilities to fabricate conventional materials under normal atmospheric conditions. Emphasis in fabrication technology has been placed on the use of sintering aids to lower the time and temperature of densification.

Hot Pressing: The hot pressing of large shaped parts is not an easy task with well-established techniques. In Fabrications Technology we have experience in this and are well underway to completing two large hot pressing systems with 100 and 350 ton presses. Our 100 ton press is complete and presently undergoing temperature profile studies to define the isothermal zone for densification. This press facility is heated inductively and will have the capability of fabricating toxic materials such as beryllium compounds. The second press could be resistively heated and used for larger parts. This facility is not operational at the time and is at the mid-point of completion.

Hot Isostatic Pressing: An unusually large and unique gas autoclave system at LLL has been fitted with a resistance furnace to deliver temperatures up to 1000°C at 50 KSI helium pressure. We have purchased a second furnace capable of reaching 1400°C. A third furnace-autoclave system surplused from ANL will allow temperatures up to 1650°C. Because the HE autoclave pressures are a factor of ten higher than conventional hot pressing and the isostatic pressing mode promotes a more uniform density, this capability is a very important tool to the fabrication of ceramics and metal-ceramic composites.

Plasma Sprayed Coatings: This capability supports several activities and tasks within the Laboratory. Its two main functions are (1) rapid application of a coating on a part, or building up to a free standing part, and (2) forming a free-flowing powder or microspheres from irregular shaped powder. The high temperature capability (30,000°K) and selective operating atmospheres (inert, hydrogen, air, etc.) enables us to handle most materials in this vacuum-tight chamber unit. Quick walk-in jobs to electrically or thermally insulate materials make up the major portion of its coating functions. Spheroidization of powder to improve flow characteristics and consolidation of powders takes up about 60% of its past functions. Minor use has been made of this unit as a heat source to test the refractory and oxidation behavior of new compounds.

Physical Vapor and Sputter Coatings: The Surface Technology Lab (STL) is concerned with advanced coating developments and applications. In the latter category STL has two sputtering systems and three evaporation systems, in addition to a toxic materials system. One of the evaporation systems is being converted for exclusive use for the deposition of plastics. The toxic materials system is in the fabrication stage and will be used for either evaporation or sputtering of nonradioactive toxic materials such as beryllium or beryllium oxide.

The emphasis of STL is upon specialized problems that are strongly affected by materials properties and materials compatibility. The combination of the Applications Facility (STL-II) with the advanced diagnostics of the Development Facility (STL-I) results in a unique capability. An example of the diagnostic capability is the 2 MeV backscattering system which allows nondestructive depth profiling.

Characterization

Principal Investigator: R. Landingham
FY 1976 Funding: \$350K

Particle Characterization: The general objectives of the Particle Characterization Analyses are to maintain and upgrade an analytical capability for characterizing and developing specifications for powders and materials. Reproducible fabrication of materials and components require logical specifications (surface area, particle size and shape, particle size distribution, and powder densities) on powders and raw materials used in their fabrication. Due to the large variety of materials used at the Laboratory, this analytical facility maintains the capability of handling toxic, radioactive pyrophoric, explosive, and hydrophilic materials as well as non-hazardous materials without cross contamination.

Physical and Mechanical Property Studies: Facilities and experienced personnel have been developed to provide physical and/or mechanical properties tests on a short-term basis on a variety of materials. These supporting tests are essential to every well organized investigation. Information reported by this facility includes phase identification by x-ray diffraction, thermal expansion, heat capacity, thermal diffusivity, differential thermal analysis, thermal gravimetric analysis, high temperature compressive and tensile creep, and mass-spec analysis.

Coating Analyses: The Surface Technology Lab (STL) has been established to combine analysis with coating development. The Advanced Development portion of the Surface Lab (STL-I) has a unique collection of process oriented diagnostic tools. Included are (1) high energy charged particle backscattering to determine coating composition profiles nondestructively, (2) high energy electron diffraction to monitor crystallite structure in the growing film, (3) two ultra-high vacuum process development systems, one coupled to each of the above diagnostic tools, (4) scanning and transmission electron microscopy and (5) various surface processing techniques such as ion etching and ion implantation.

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Sandia Materials Program

ORGANIC MATERIALSMechanical Properties of Polymers

Principal Investigator: R. G. Kepler

FY 1976 Funding: \$450K

Theoretical and experimental methods are being developed for the prediction of the long-term mechanical properties of bulk polymers. The theoretical studies include studies directed toward understanding of the relationships between bulk properties and molecular structure, and methods for relating chemical and physical relaxation to bulk mechanical and thermal properties. Experimental methods for the determination of moduli, creep and stress relaxation, chemically induced bulk property change, plasticization and permeation by gases, and morphology are being developed. These experimental and theoretical techniques will allow more confident prediction of long-term properties of both rigid and nonrigid polymeric materials.

The technologies influenced by these studies are: encapsulation, electrical insulation, thermal protection and regulation, mechanical shock mitigation, environmental sealing and control, and mechanical design.

Theoretical techniques have been developed to predict: (1) Gruneisen parameters for polymers over the temperature range from absolute zero to decomposition temperatures based on a cell model for amorphous polymers; (2) equations of state for amorphous materials; and (3) the influence of network structure on relaxation in crosslinked systems. A computer simulation of multiple chain systems has been developed to assist in the understanding of the effect of interchain interactions on mechanical properties.

Experimental methods for the evaluation of the various parameters necessary for complete specification of mechanical response to a variety of external stimuli are being developed. These involve moduli determination, chain relaxation studies using NMR and fluorescence spectroscopy, thermomechanical analysis, differential thermal analysis, and long term mechanical stress relaxation measurement.

Chemistry of Organic Materials

Principal Investigator: P. I. Kepler

FY 1976 Funding: \$350K

The chemistry of organic materials is being studied to provide a base for materials selection and materials engineering for such applications as photo and electron beam masking resists, radiation tolerant organic hydrogen getters, radiation dosimeters, photo and radiation stable plastics, photo-sensitive polymers for data storage, special materials for weapons application, and thermally stable thin film insulators. Syntheses, reaction mechanism studies, and reaction kinetic studies are now underway.

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The reaction mechanisms and kinetics of thermal and radiation decompositions are being studied to provide a data background for materials choice as (1) thermally stable dielectric coatings, (2) thermally stable and radiation tolerant encapsulants, (3) positive and negative photoresists, and (4) radiation and thermally stable hydrogen getters.

Synthetic efforts include the synthesis of special polymers for mechanical property studies, photo and radiation sensitive polymers for resist applications, special monomers for high-temperature insulation, model compounds for reaction chemistry studies, and diacetylenes for use in reactive gas gettering operations.

The technologies influenced are encapsulation, microelectronics, reactor safety, environmental control, medical electronics, and synthetic chemistry of polymers.

The approach taken is to synthesize model compounds, determine their reactivity toward hostile or reactive environments (both mechanisms and kinetics) and, after establishing relationships between structure and behavior, synthesize stable (or reactive special-use materials).

Electronic Behavior of Organic Materials

Principal Investigator: P. I. Kepler

FY 1976 Funding: \$500K

The electronic behavior of organic materials is being studied both to increase our understanding of the relationships between materials structure and such electronic properties as dielectric constant and loss, polarization (electrets, ferroelectrics), charge carrier production and transport, high-voltage breakdown and ionic conduction and to provide an expanded data base on these properties for the designer.

Technology areas affected include particle acceleration design, fusion reactors, sensors and transducers, electrical insulation, radiation hardening, electrooptical devices, electrochemical detectors, and power generation and support.

The approaches taken are the study of time and field dependence of dielectric breakdown (bulk and surface), the study of the field dependence photoinduced charge carrier production, the study of the time evolution of photoconduction, the study of piezoelectricity and pyroelectricity in polymers, the study of structural features of solutions of charged particles and charged bacteria by light scattering (static and dynamic), and the study of the structure and transitions in liquid crystals.

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In this program, theories of charge carrier production in polymers and organic crystals have been studied and a coherent picture of carrier production and transport has been developed. Further studies are underway to relate structural features of the material to the pertinent transport and production parameters. Ferroelectric polymers are being studied to provide new materials for inexpensive detectors and transducers. The mechanism of surface breakdown initiation at high electric fields has been elucidated and future studies are directed toward understanding the sustaining phase in detail.

The structure of solutions of charged macromolecular particles are being studied both dynamically and statically by light scattering techniques. Theories for solution behavior will be developed, and liquid crystal properties will be investigated by these same techniques.

Sandia Materials Program

INORGANIC MATERIALS

Principal Investigator: J. E. Schirber

FY 1976 Funding: \$12,000K

The object of this program is to strengthen the Laboratories' technological position in inorganic materials and processes involving these materials for present and potential application to weapons and energy-related problems. This technological position is achieved by a combination of experimental and analytical or theoretical approaches and characterization which lead to a sufficient understanding of the controlling factors so as to permit optimization of the material for the given application. For example, typical goals for device applications include long life, high reliability, small size and hardness in extreme environments involving blast, shock and radiation. The sciences brought to bear on these problems include solid state, surface and device physics, physical and analytical chemistry, metallurgy and ceramics and computational science.

Major areas of emphasis include H_2 and He in materials, solid electrolytes, electrical and mass transport in insulators, semiconductors and amorphous materials, surface analysis and modification, radiation effects in semiconducting and insulating materials, permeation and diffusion in glasses and ceramics, shock-activated phenomena in ferroelectric and ferromagnetic materials, studies of explosives and pyrotechnic materials and equation of state and phase changes in a variety of substances, including soil and rocks.

H_2 and He in Materials: Percolation and electronic band theory have been coupled with nuclear magnetic resonance and other microscopic probes such as nuclear reaction spectroscopy to propose a predictive model for 3He release in metal tritides; 3He implantation has been used to simulate release.

Surface and Interface Modification and Analysis: Ion implantation is used as a process to modify and dope materials. Ion and damage profiles for implanted ions in semiconductors have been measured and theoretical understanding developed. Charge exchange at surfaces, at contacts and with respect to corrosion, chemisorption, diffusion and solubility have been studied and provide the basis for advances in silicon-integrated and other circuit technology.

Defects in Solids: Mechanisms for production and the identification of irradiation-produced defects in bulk and at surfaces and their effect on charge storage and motion in MOS or MNOS devices have been studied. The relation of both chemical and irradiation-produced defects to mechanical properties of materials are considered with goals of understanding H embrittlement and defect microstructure.

Solid Electrolytes: Mass and electrical transport in superionic materials which have potential for solid-state batteries are under study. The nature of electrode surfaces and electrochemical reactions are receiving attention. An increase in understanding of the electrochemical and chemical mechanisms is needed to improve reliability and energy density of 60-70-minute thermal batteries.

Shock- and Temperature-Activated Device Materials: The response to stress of ferroelectric, piezoelectric, and ferromagnetic materials is studied with the aim of improving materials for firesets, neutron generators, and pulse power supplies. An example includes attempts to extend the utility of lithium niobate for 100 kV power supplies.

Explosives: Initiation and detonation criteria in heterogeneous secondary explosives such as 9404 and TATB are being determined with emphasis on effects of particle size, charge density, and sensitizers. Studies are underway of deflagration-to-detonation transition phenomena.

Phase Changes and High Stress Equation of State: Extensive shock wave and energy deposition studies of polymorphic, melting and vaporization phase change kinetics are underway on a variety of materials. These studies are relevant to blow-off phenomena and explosive components used in weapons technology.

Earth Material: Studies of "fissurization" models of rock and soils will allow prediction of dilatency, fracture and fragmentation. These models are required for support of earth penetrator weapons technology.

Glass Ceramics: Ease of fabrication and high-temperature stability make these materials attractive for weapons applications. Experimental investigation of relationships between physical properties and composition and microstructure provide a basis for "tailor-making" glass-ceramic materials for specific applications. Research on the crystallization process is expected to yield new materials with oriented structure and new properties.

Sandia Materials ProgramMETALLURGY

There are three major areas of metals research which though described separately are strongly interrelated. These projects are also dependent on and contribute to the materials characterization and inorganic chemistry program. A strong interdependency of Sandia's materials research has been created by the stringent demands placed on materials in nuclear weapons by operational requirements and long storage times. The Sandia metallurgical program therefore emphasizes the synergistic effect of alloy chemistry, microstructure, processing, and surface conditions on corrosion, mechanical, chemical, and physical properties.

Structural Metals

| Principal Investigator: M. J. Davis
FY 1976 Funding: \$2400K

Structural metals program is based on specialized techniques to determine the behavior of metals mechanically and chemically. Fracture, deformation, and compatibility studies have made use of experimental techniques to separate variables such as constant stress intensity stress corrosion specimens tested in controlled environments ranging from molten salts to 1 torr hydrogen gas while monitored by acoustic emission. These projects are supported by a unique melting, electrodeposition, powder metallurgy, and joining capability. Specific FY 76 programs were:

Experimental and theoretical studies of the effects of combinations of creep, creep-fatigue, and monotonic loading on subsequent mechanical response are underway. Detailed experiments are being conducted on the thermal fatigue of solder alloys utilized in encapsulated printed circuit boards. A constitutive relation has been found which will allow prediction of cyclide loading behavior from monotonic experiments. Attempts will be made to apply this relation to more complex histories.

- Experimental and theoretical studies of polymorphic melting and vaporization phase changes and their kinetics are underway to characterize loss of strength mixed phase states and vapor pressure under extreme conditions of pressure or temperature.

- Studies of low temperature effect on hydrogen embrittlement of normally compatible metals are the emphasis of the extensive hydrogen containment program.

- Advanced fracture mechanics techniques are being used to characterize and develop ultra high toughness alloys. Unique experimental development by Sandia in FY 76 has stimulated this program.

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- Alloy development is supported by unique heavily instrumented VAR and VIM furnaces which will allow heretofore unavailable control of ingot chemistry and macrosegregation plus a level of understanding which will permit efficient transfer of the technology to production agencies. Ultra high strength maraging steels have been produced and are being studied for forgeability. Corrosion in systems with porous metallic coatings is being modeled and techniques to evaluate porosity are being developed.

- The corrosion and stress corrosion of alloys are being studied. It is controlled by a strong interdisciplinary program between surface, physical, mechanical, and process metallurgy with organic, inorganic, and electro chemistry.

The general corrosion and localized corrosion behavior, including pitting, stress corrosion cracking, and galvanic corrosion of alloys are being studied. The research is aimed at elucidating the role of metallurgical, chemical, and mechanical parameters, and ultimately at prevention techniques.

- The relationship between heat treatment, weld practices, and microstructure on the mechanical behavior of moderate strength steels and titanium alloys is being investigated. Specific concerns are the effects of temperature and strain reaction, anisotropic tensile behavior, fracture toughness, and notch sensitivity.

- Moderate strength uranium alloys are being developed with improvised fracture toughness and resistance to environmental degradation.

- High temperature metallographic reactions are being studied and applied toward denying special nuclear materials and the study of the chemical interactions observed during a hypothetical reactor meltdown.

Component Metals

Principal Investigator: M. J. Davis
FY 1976 Funding: \$1600K

The component metals program emphasizes the development of special or multiple purpose alloys and appropriate processing. Mechanical deformation, joining, compatibility, and electrical behavior of alloys in complex environments are studied. Specific FY 76 programs were:

- Studies are underway to improve ductility and welding of molybdenum used in neutron generators. The metallurgical factor influencing ductility of FCC metals are being studied.

- Studies of stress relaxation and delayed failure under constant load of both precious and base metal springs are directed at predicting long-time elastic behavior. Improved alloy compositions and processes are sought through delineation of the deleterious microstructural elements in extant alloys.

- New palladium alloys are being studied to provide a controlled means of removing hydrogen from systems.

- Microelectronic metal research emphasizing thin and thick film conductors and resistors is underway to determine transport, adhesion, bonding, and aging

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behavior, as well as the effects of EMP and radiation on performance.

Surface Metallurgy

Principal Investigator: M. J. Davis

FY 1976 Funding: \$1200K

This project is concerned with understanding the metallurgical properties of surfaces used in weapons development and the generation of desired surface properties by choice of materials, surface modification, and the forming of new surfaces. Emphasis is on bridging the gap between basic studies and engineering applications. Specific areas were:

- Implantation metallurgy. Ion implantation and ion backscattering are being used to modify materials in a controlled manner and determine basic metallurgical properties.

- Surface deformation. Studies of surface-surface contact deformation and adhesion using carefully controlled environmental conditions and sensitive measurement techniques are applied to friction and wear, electrical contacts, and deformation bonding. Analytical techniques include laser interferometry, acoustic emission, contact resistance measurements, coefficient of adhesion measurements, and microshear testing.

- Electrical contact arcing studies involve the effect of voltage on contact erosion, effects of contaminants on contact resistance, and material transfer between arcing contacts applied to electrical contact design and in the development of nondestructive testing procedures.

- Thin film and coating development is directed at obtaining desirable surface properties by depositing new surfaces using vacuum deposition, sputter deposition, ion plating, electroplating, and chemical vapor deposition techniques.

- Gas-metal reaction research is directed at the effect of hydrogen absorption on the mechanical properties of surfaces, effect of surface condition on hydrogen absorption and permeation. Hydrogen-metal phase relationships at high pressures and temperatures, and diffusion and thermal desorption of gases in metals is also being studied.

- Surface cleaning and contamination. Cleaning by sputter cleaning, reactive plasma cleaning, solvent cleaning, UV/O₃ cleaning, contamination detection using ⁸⁵Kr, contact angle analysis, and soft x-ray appearance potential spectroscopy.

Sandia Materials Program

COMPOSITE MATERIALS

Principal Investigator: O. M. Schuster
FY 1976 Funding: \$1000K

This program is based upon those disciplines needed to fully understand and characterize high-performance, fiber-reinforced composites. The results of continuing research efforts in fabrication and processing techniques, static and dynamic mechanical behavior and structural analysis, fracture mechanics and viscoelastic effects are being applied to several weapon programs. Composites being investigated incorporate both resin and metal matrices reinforced with a variety of high-strength materials: boron and Borsic filaments; Kevlar, glass, and graphite yarns and cloths; ceramic whiskers and filaments.

The well-established technologies listed above have been used to develop a variety of prototype weapon parts: e.g., boron-reinforced aluminum deployable bomb fins, a thick-walled cylindrical artillery shell ring stiffener and thin-walled conical reentry vehicle shapes; and graphite or Kevlar filament-reinforced resin stiffeners for electronic components, radar and telemetry systems. Two specifically applied efforts are a thorough characterization program studying Kevlar and nylon filaments for parachute applications and the development of alumina-cylinder loaded resins for weapon protection systems.

The current plan is to maintain our basic capabilities while gradually expanding into the areas of joining technology and environmental stability of composites.

Sandia Materials Program

CHARACTERIZATION TECHNOLOGY

Principal Investigator: F. L. Vook
FY 1976 Funding: \$12,500K

This program is concerned with the development and application of unique and state-of-the-art techniques for the detailed characterization of a broad spectrum of materials, including inorganic, organic, metallic, and composite materials. The characterization areas include: surface and near-surface analysis; composition, electronic and crystal structure; thermophysical, chemical, and electrochemical properties; viscoelastic, shock, high pressure, and mechanical properties; radiation and defect properties; magnetic, superconducting, and transport properties. Emphasis is placed on weapon and/or energy-related materials having extended life, ultrahigh reliability, and the ability to withstand extreme environments such as blast, shock and radiation. New techniques and facilities are being developed to selectively characterize materials and simulate environments to increase command and control, safety, capability and reliability.

Major Technical Emphasis in FY 76Ion Solid Interactions

Van de Graaff accelerators (2.5, 2.0, and 0.4 MeV), two 0.3 MeV heavy-ion implanters and an 80-keV high current heavy-ion accelerator operate at ultrahigh vacuum and are equipped with temperature controlled target stages and precision goniometers for ion implantation and channeling measurements. An on-line transmission electron microscope has been integrated with a 2.5-MeV Van de Graaff and 80-keV heavy-ion accelerator for in situ implantation and analysis. A new 4 MeV tandem accelerator is planned for FY 76. Helium and hydrogen lattice locations, diffusion, solubility, damage, and depth distributions are characterized by ion backscattering, channeling and ion-induced nuclear reaction analysis. These studies are applicable to CTR and hydrogen economy research and development, the simulation of neutron damage in reactor materials, and alloy and corrosion science.

Surface and Near-Surface Analysis

Near-surface ion beam analysis is used as a nondestructive technique for determining composition variation with depth in micron thick layers. Current techniques include ion backscattering, ion nuclear reaction microanalysis, ion-induced x-rays, secondary ion emission. Near-surface characterization also includes SEM, TEM, x-ray structure, and electron microprobe analysis. Surface characterization techniques include soft x-ray appearance potential spectroscopy, binary scattering of noble gas ions, ion microprobe, thermal and field desorption and field ion microscopy, low-energy electron diffraction, characteristic ionization and loss spectroscopy, Auger, ESCA, and photoelectron spectroscopies. A unique gated field desorption microscope has been invented

and applied to the imaging of hydrogen chemisorption and field ionization states on metal crystallographic surfaces. These instruments are applicable to CTR studies of surface-induced contamination of plasmas as well as to fundamental catalysis studies, catalyst characterization, corrosion science, and reactor materials damage.

Radiation Response

Extensive facilities are available for producing and characterizing the radiation effects on materials by neutrons, ions, electrons, x-rays, and gamma rays. Five Van de Graaff accelerators, three heavy-ion accelerators, two pulsed reactors, three pulsed relativistic electron facilities, pulsed laser, and pulsed x-ray facilities are used with in situ optical spectrometers, calorimetric systems, and low-temperature irradiation and neutron radiography capabilities. Transient radiation effects are studied for military applications and pulsed fusion CTR research. Flash x-ray facilities can be used for transient reactor materials and component behavior and safety analysis.

Defects

Electron paramagnetic and nuclear magnetic resonance, positron annihilation, infrared absorption, light scattering, photoluminescence, depth resolved catholuminescence, ultrasonics, cantilever beams, stress measurements, electrical techniques and ion implantation are used to identify and characterize defects in solids. These techniques are applicable to reactor materials damage and photovoltaic materials.

Mechanical Response

Techniques to characterize the response of materials under high rates of energy deposition including melting phenomena in 10^{-6} sec or less have been developed to study the behavior of shock compressed materials. This is complemented by a complete static high pressure laboratory. Major characterization techniques are: submicrosecond resolution quartz and lithium niobate gages, displacement and velocity interferometric systems, in-flight stereo and holographic recording techniques, and high-speed photography. Major facilities include: two-stage light-gas/power gun facilities, compressed gas gun facility, triaxial servo control ultrastiff machine with capabilities of 1 GPa pressures and 1.78 million Newtons force rating, and an ultrasonics facility producing sinusoidal modes with concurrent temperatures and pressure control. Characterization of deformation, creep, stress relaxation, and fracture of metals and ceramics under cyclic loading is being investigated and is directly related to power reactor safety studies. Hydraulic jets both continuous and pulsed have been characterized for applications ranging from coal mining and rock tunneling to the machining of metals. Techniques to characterize shock wave phenomena have been used to analyze the safety of containers for radioactive materials, reactor safety analysis, fracture behavior of anisotropic oil shales, the development of earth-penetrating projectiles, rock drilling and blasting, and oil shale retorting.

High Temperature Thermal Properties

Techniques have been developed to determine enthalpies, heat capacity, thermal diffusivities and conductivities and equation of state of solid, liquid and gaseous materials to temperatures in excess of 6000K and 10 MPa. Electromagnetic levitation, liquid argon calorimetry, and laser heating are used to study stainless steels, metal hydrides and refractory liquids. These techniques are applicable to reactor safety, nuclear waste disposal, coal gasification, oil shale extraction and solar energy studies. Two plasma jets (2.5 Megawatt and 160 KW) are used to characterize parallel ablation and erosion of refractory materials in a channel flow device. Optical techniques have been developed to measure heavy particle and electron temperature, plasma velocity, electron density, and total gas enthalpy. These facilities are used to characterize heat shields and antenna window materials for reentry vehicles and can be used for MHD channel erosion studies.

OBJECTIVES

Establish a correlation among alloy corrosion rates and gas composition, temperature, pressure, and the level of major alloying elements in alloys to be used for internal components of coal gasifiers

PROJECT JUSTIFICATION

Since 1972, a program has been carried out by the Metals Properties Council (MPC) to discover materials suitable for use in coal gasification plants. Results of the Phase I laboratory testing of this program have indicated that only a very few alloys are able to successfully withstand the corrosive action of the coal gasification atmosphere at 1500°F if H₂S is present in the gas mixture. The most promising alloys are those with high chromium and nickel contents; a 50 Cr-50 Ni alloy is the most corrosion-resistant alloy so far.

Unfortunately, preliminary results in pilot plants have not confirmed laboratory tests in all cases. Exposure tests in the regenerator of the CO₂ acceptor plant have shown that very severe corrosion of the 50 Cr-50 Ni alloy and significant corrosion of other high chromium alloys occur. This indicates that differences in gas composition, temperature, and pressure may markedly alter corrosion test results but, at this moment, we do not know how.

This project will be of great value in predicting the behavior of materials in proposed gasification processes with widely varying atmospheric compositions and in increasing our fundamental understanding of metal corrosion in gasifier atmospheres. It will establish a firm correlation between corrosion rate and operating pressure, thereby enabling the development of atmospheric pressure corrosion tests which will greatly simplify and accelerate routine corrosion testing of candidate materials for coal gasification applications. In addition, the test results will be of considerable value in correlating the laboratory and in situ pilot plant tests of the MPC program and in explaining discrepancies.

CONTRACT DATA

CONTRACT NO. W-7405-ENG-92-Task 92
CONTRACTOR Battelle Memorial Institute
ADDRESS 505 King Avenue
 Columbus, Ohio 43201
PRIN. INVEST. I. G. Wright
BEGIN/END 1 May 1976 thru 28 Feb 1979
CONTRACT VALUE \$244,000
SPONSOR FER
DIRECTORATE Materials and Power Generation
TECH. PROJ. OFF. W. T. Bakker
CATEGORY/DETAIL Materials and Components/Materials

FUNDING
 (thousands of dollars)

| FY | ERDA | CONTRACTOR | TOTAL |
|----|------|------------|-------|
| TQ | 100 | -- | 100 |

DESCRIPTION

This materials testing project consists of five tasks to:

- I. Statistically design a test procedure to expose selected alloys to various coal gasification atmospheres at temperatures of 1500 to 2000°F and pressures of 500 to 1500 psi.
- II. Prepare suitable test specimens from the alloys. Expose two samples of each alloy simultaneously to increase the reliability of the test results. Completely characterize the metal stock from which the samples are taken through chemical and microstructural analyses and tensile and hardness testing.
- III. Conduct exposure tests at the temperatures, pressures, and gas compositions required by the factorial design. Expose the specimens to a continuous gas flow with the gas atmosphere being completely changed at least once every 30 minutes. Use a minimum exposure time at least equal to that time required to produce significant corrosion on 316 stainless steel using the MPC gas composition.
- IV. Characterize fully all specimens after exposure. Photograph them and measure their weight change. Measure the scale thickness together with corrosive penetration and thickness of the remaining sound metal. Use the SEM and the microprobe as necessary to identify corrosion products. Perform tensile and hardness tests on the specimens after exposure.
- V. Determine the statistical correlations between the experimental variables and the corrosion rate.

ACTIVITY SCHEDULE

| ACTIVITY | FY 76 | TRANS | FY 77 | FY 78 | FY 79 | FY 80 | FY 81 |
|--------------------------------|-------|-------|-------|-------|-------|-------|-------|
| DESIGN TEST PROCEDURE | | | | | | | |
| PREPARE ALLOY SPECIMENS | | | | | | | |
| PERFORM TESTS | | | | | | | |
| CHARACTERIZE EXPOSED SPECIMENS | | | | | | | |
| CORRELATE RESULTS | | | | | | | |

Correlation of High-Temperature Corrosion Behavior of Structural Alloys in Coal Conversion Environments with Components of the Alloys and of the Corrosive Environments

03-1

Coal/Materials and Components/Materials Corrosion Behavior of Alloys

Provide design and reliability information on selected gas turbine nozzle and bucket materials to define firm development needs for improved component materials and coatings, using gas environments that are representative of a combined-cycle central station generating plant burning coal-derived low-Btu gas and coal-derived fuel oil.

PROJECT JUSTIFICATION

Very little data are available on nozzle and bucket life of gas turbines used in combined-cycle central station generating plants when exposed to fuels with high alkali metal content. General Electric will define potential problems using coal-derived low-Btu gas and low-sulfur liquid fuel with different impurity levels. Test materials will be selected from those currently available and in advanced development. The program will provide a selection of coal feedstock and the selection rationale, a listing of tested nozzle and bucket materials, an evaluation that identifies expected gas turbine critical parts life with a variety of contamination levels, and an evaluation that identifies the program's impact on gas turbines, coal-derived processes, and clean up systems.

This program will interface with and benefit from a complementary, cooperative program between General Electric and ERDA's Morgantown Energy Research Center's (MERC) low-Btu gas facility using both high-sodium and low-sodium content coals.

CONTRACT NO. E(49-18)-1765
CONTRACTOR The General Electric Co.
ADDRESS 1 River Road
Schenectady, N. Y. 12345

PRIN. INVEST. S. M. Kaplan
BEGIN/END 26 Jun 1975 thru 30 Jun 1977

CONTRACT VALUE \$1,073,029

SPONSOR FER/CCU

DIRECTORATE Materials and Power Generation

TECH. PROJ. OFF. J. Smith

CATEGORY/DETAIL Materials and Components/Materials

FUNDING (thousands of dollars)

| FY | ERDA | CONTRACTOR | TOTAL |
|----|------|------------|-------|
| 75 | 1024 | — — | 1024 |
| 76 | 49 | — — | 49 |

This program is divided into the following seven tasks.

- I. Select test materials from those currently used in industrial gas turbines and from those in advanced development. Define operating conditions and identify materials with adequate surface stability and mechanical properties.
- II. Select two to four coal feedstocks and define their characteristics, particularly alkali metal content, pertinent to gas turbine durability. Important impurities include sodium, potassium, vanadium, lead, chlorides, nitrogen, and sulfur.
- III. Establish test conditions for the initial tests based on the temperature, velocity, and other design considerations representative of present and advanced generation open-cycle industrial gas turbines operating on a low-Btu gaseous fuel and a low-sulfur, coal-derived oil. Base screening test parameters on fuel characterization derived from the selected feedstocks and the results of a thermodynamic analysis and the initial tests. Base confirmation test parameters upon the results of all previous testing and include preferred materials, cooling design, and representative fuel cleanup levels.
- IV. Design and procure test specimens for the initial, screening, and confirmation tests at existing GE and ERDA test facilities.
- V. Conduct initial tests to define hot corrosion, erosion, fouling, and other potential problems using coal-derived liquid fuels from two coal feedstocks with different impurity levels. Perform screening tests on approximately 20 selected materials using environments determined in Tasks II and III. After exposure, cross-section and microscopically examine specimens to measure corrosion penetration.
- VI. Evaluate initial test results using a GE model to predict the life of turbine buckets and nozzles in order to select the cooling design for the confirmation test. Recommend state of the art corrosion inhibitors, ash modifiers, and internal cleaning procedures. Consider tradeoffs among gas cleanup, gas turbine design, and operating procedures leading to life improvement.
- VII. Conduct confirmation tests using a low-sulfur, coal-derived oil and a low-Btu gas and test samples from Task IV.

ACTIVITY SCHEDULE

| ACTIVITY | FY 76 | TRANS | FY 77 | FY 78 | FY 79 | FY 80 | FY 81 |
|-----------------------------------|-------|-------|-------|-------|-------|-------|-------|
| SELECT TEST MATERIALS | START | | | | | | |
| SELECT FEEDSTOCKS | START | | | | | | |
| DEFINE TEST CONDITIONS | START | | | | | | |
| DESIGN AND PROCURE TEST SPECIMENS | START | | | | | | |
| CONDUCT INITIAL (I) TESTS | START | | | | | | |
| PERFORM SCREENING (S) TESTS | | | (I) | (S) | | | |
| EVALUATE TEST RESULTS | | | | | | | |
| PERFORM CONFIRMATION TESTS | | | | | | | |

OBJECTIVES

Test available metal and ceramic construction materials, primarily in several pilot plants, for service under the hostile conditions existing in coal gasification equipment

PROJECT JUSTIFICATION

In 1972 it became clear that coal gasification pilot plant problems could only be solved by materials testing and research to ensure that satisfactory materials would be available for commercial coal gasification plant components. A Metals Properties Council (MPC) Working Group formed to review the Bi-Gas, Hygas, and CO₂-Acceptor processes in the area of materials corrosion/erosion determined that for all three processes:

- Potential metal problems included sulfur corrosion, carburizing, nitriding, creep, and erosion
- Refractories presented a materials problem
- Common corrosion problems existed in quenching, scrubbing, and pretreating areas in addition to valves and transfer lines
- Unique corrosion and erosion problems existed for all three processes

The American Gas Association (AGA) initiated this program in 1972 with the MPC with the understanding that the Office of Coal Research (OCR) (the precursor of the ERDA Office of Fossil Energy) would cosponsor the program when funds became available. In 1974, the OCR determined that it should provide all project funding starting in FY 1975 because of the expanding scope of the project. This project consists of five phases. The first four are being performed by the Illinois Institute of Technology Research Institute (IITRI). The fifth phase has been awarded to the Southwest Research Institute (SRI). (See page 03-4 for a description of the SRI effort.)

A modification, which extends the IITRI Phase II effort, is necessary because the work accomplished to date by IITRI has shown deviations between laboratory corrosion tests using standardized, simulated process conditions and actual exposures in pilot plants. These differences indicate that variations in operating conditions between various coal gasification processes are large enough to cause significant differences in corrosion behavior. Since it is not known at this time which processes will be commercialized in the future, it is necessary to expose materials samples in all existing and future pilot plants.

CONTRACT DATA

CONTRACT NO. E(49-18)-1784(Mod 2)
CONTRACTOR Metals Properties Council, Inc./
Illinois Inst. of Technology, Research Inst.
ADDRESS 10 West 35th Street
Chicago, Ill. 60616

PRIN. INVEST. A. Schaeffer/M. Howes

BEGIN/END 26 Jun 1975 thru 31 Dec 1979

CONTRACT VALUE \$2,710,000

SPONSOR FER

DIRECTORATE Materials and Power Generation

TECH. PROJ. OFF. Dapkunas (I,IV) / Bakker (II) / Cox (III)

CATEGORY/DETAIL Materials and Components/Materials

FUNDING (thousands of dollars)

| FY | ERDA | CONTRACTOR | TOTAL |
|----|------|------------|-------|
| 75 | 2060 | -- | 2060 |
| 76 | 500 | -- | 500 |

PROJECT DESCRIPTION

The IITRI portion of this MPC materials study consists of the following four phases covering both metals and ceramic materials for use in coal gasification plants.

Phase I - High Temperature Corrosion - perform laboratory corrosion testing of various candidate alloys for gasifier internal components in simulated coal gasification atmospheres at elevated temperatures and pressures.

Phase II - Pilot Plant Exposure - evaluate metallic and ceramic materials exposed to various locations in operating pilot plants (Hygas, Bi-gas, CO₂, Acceptor, Synthane, Battelle, and IGT-steam-iron processes).

Phase III - Aqueous Corrosion Testing - conduct laboratory corrosion testing of alloys in aggressive environments simulating quench systems in coal gasification plants.

Phase IV - Erosion/Corrosion Testing - perform laboratory erosion/corrosion testing of metals and refractories at high temperatures and pressures in coal gasification atmospheres.

ACTIVITY SCHEDULE

| ACTIVITY | FY 76 | TRANS | FY 77 | FY 78 | FY 79 | FY 80 | FY 81 |
|----------------------------|-------|-------|-------|-------|-------|-------|-------|
| HIGH TEMPERATURE CORROSION | | | | | | | |
| PILOT PLANT EXPOSURE | | | | | | | |
| AQUEOUS CORROSION TESTING | | | | | | | |
| EROSION/CORROSION TESTING | | | | | | | |

Expansion of Phase II Corrosion Testing of Metal and Refractory Samples in Gasification Pilot Plants

03-3

Coal/Materials and Components/Materials Corrosion Testing

OBJECTIVES

Generate engineering data, including both physical and mechanical properties in the coal gasification environment, on materials that are the most likely candidates for use in the construction of plants for the gasification of coal

PROJECT JUSTIFICATION

Exposure of materials to the hostile environments encountered in coal gasification plants can seriously affect their mechanical properties. No data are available detailing the mechanical properties of candidate alloys in the coal gasification atmosphere nor does information exist as to how those properties are altered by prolonged exposure to the atmosphere. These data are required before large, production-size plants can be designed to operate for long periods of time free from mechanical failures. Consultation with representatives of several large engineering firms that specialize in the construction of large gas facilities has confirmed the necessity of generating these data.

Research on "Materials Research for Coal Gasification" has been conducted by the Metals Properties Council (MPC) since October 1972. The program consists of five phases: high temperature corrosion, pilot plant exposures, aqueous corrosion, erosion/corrosion, and engineering properties.

The philosophy of the program as initially designed was to use the first four phases as a screening mechanism to determine those alloys that are likely candidates for use in equipment in production-size coal gasification plants. Those alloys surviving the screening process would then be considered in the final phase where extensive engineering data of mechanical and physical properties would be compiled for their behavior in the coal gasification environment. If insufficient data existed, then MPC would devise a research program to develop the needed data. The program has progressed to the stage where the number of candidate materials has been reduced to a reasonably small number and, since the search for existing data has produced none, it is now necessary to begin developing engineering data on remaining materials.

Physical and Mechanical Properties of Certain Alloys (Phase V of "Materials Research for Coal Gasification")

CONTRACT DATA

CONTRACT NO. E(49-18)-1784 (Mod 3)
 CONTRACTOR Metals Properties Council, Inc./
 Southwest Research Institute
 ADDRESS P.O. Drawer 28510
 San Antonio, Tex. 78284
 PRIN. INVEST. A. Schaeffer/C. H. Wells
 BEGIN/END 1 Jul 1976 thru 30 Jun 1979
 CONTRACT VALUE \$1,913,289
 SPONSOR FER
 DIRECTORATE Materials and Power Generation
 TECH. PROJ. OFF. T. B. Cox
 CATEGORY/DETAIL Materials and Components/Materials

FUNDING
 (thousands of dollars)

| FY | ERDA | CONTRACTOR | TOTAL |
|----|------|------------|-------|
| 76 | 1000 | -- | 1000 |
| TQ | 140 | -- | 140 |

PROJECT DESCRIPTION

This materials research project consists of two tasks to:

- I. Conduct a full set of mechanical tests in air to serve as a base line for the data developed in the MPC Coal Gasification Atmosphere (CGA). Pre-expose specimens to the CGA at 1000 psi for 1000 hr at temperatures of 1200, 1500, and 1850°F. Conduct subsequent tests at temperatures of 80, 1200, 1500, and 1850°F and in air and/or CGA atmospheres depending on the particular test. All tests are to be carried out in accordance with applicable ASTM specifications using standard specimens, unless otherwise directed. The mechanical tests to be carried out include tension, hardness, impact, stress rupture, and fatigue.
- II. Determine the following physical properties by established methods for each material: melting point, specific heat, density, and emissivity. Measure the following properties from room temperature to 2000°F: thermal expansion, thermal conductivity, modulus of elasticity, and Poisson's Ratio.

Materials to be tested include Incoloy-800, type 310 stainless steel, HK-40, and 50Cr-50Ni. Selected data is to be determined for these alloys for welded joints (conforming to AWS and ASME standards) in addition to the alloy itself. Mechanical and physical data on the materials only are to be obtained for types 309 and 314 stainless steel, RA-330, and HK-40-3Si.

ACTIVITY SCHEDULE

| ACTIVITY | FY 76 | TRANS | FY 77 | FY 78 | FY 79 | FY 80 | FY 81 |
|-------------------------------|-------|-------|-------|-------|-------|-------|-------|
| CONDUCT MECHANICAL TESTS | | | | | | | |
| DETERMINE PHYSICAL PROPERTIES | | | | | | | |

OBJECTIVES

Test, identify, and evaluate commercial and advanced experimental materials and coatings under design conditions simulating advanced fuel power cycle combinations

PROJECT JUSTIFICATION

Advanced power systems will operate at higher temperatures than current systems in order to improve power conversion efficiency. Materials that will be able to operate reliably at these higher temperatures need to be identified. Corrosion data must be developed to show how well these materials will hold up in the advanced power system environments. This project will produce data to supplement data being developed in other Fireside Corrosion tasks.

CONTRACT NO.

E(49-18)-2045

CONTRACTOR

Combustion Engineering, Inc.

ADDRESS

1000 Prospect Hill Road
Windsor, Conn. 06095

PRIN. INVEST.

A. L. Plumley

BEGIN/END

30 Jun 1975 thru 30 Jun 1977

CONTRACT VALUE

\$937,471

SPONSOR

FER/CCU

DIRECTORATE

Materials and Power Generation

TECH. PROJ. OFF.

S. J. Dapkunas

CATEGORY/DETAIL

Materials and Components/Materials

FUNDING
 (thousands of dollars)

| FY | ERDA | CONTRACTOR | TOTAL |
|----|------|------------|-------|
| 75 | 937 | -- | 937 |

PROJECT DESCRIPTION

This project to test materials for use in advanced power cycles consists of eight tasks to:

- I. Select four high temperature alloys for 300 hr tests at 1200 to 1650°F in the Kreisinger Development Laboratory (KDL) facility. Evaluate at least two coatings for further testing on these alloys.
- II. Evaluate four coal feed stocks, which represent realistic ranges of feed stocks available for power generation in the U.S., in the KDL test furnace and in operating utilities.
- III. Establish test conditions to be used in further testing in the KDL solid fuel-fired test furnace aimed at simulating actual field units burning the selected coal feed stocks.
- IV. Establish test set ups in the KDL furnace so that the probes will be exposed to the environment and gas velocity in a geometric arrangement similar to the superheater section of actual boilers for liquid-coupled Rankine and closed Brayton cycle systems.
- V. Fabricate test sections of each material to be included on probes from the same stock lot of commercially available tubing material. Retain original unexposed tubing to allow comparison of properties of materials showing changes resulting from exposure in furnace environments.
- VI. Test specimens in KDL furnace and operating utility boilers at metal surface temperatures of 1250 to 1850°F (controlled by regulating amount of air passed through test specimens) and two gas temperatures. Determine loss of metal by measuring weight loss and reduction in wall thickness of test specimens.
- VII. Evaluate the results of the testing on the basis of changes occurring in the samples as a result of exposure to the test environment.
- VIII. If test results indicate that a repeat of a particular test or an extension of testing is necessary, perform the necessary tests using original feed stock samples.

ACTIVITY SCHEDULE

| ACTIVITY | FY 76 | TRANS | FY 77 | FY 78 | FY 79 | FY 80 | FY 81 |
|---------------------------|-------|-------|-------|-------|-------|-------|-------|
| SELECT MATERIALS | - | | | | | | |
| SELECT COAL FEEDSTOCKS | - | | | | | | |
| ESTABLISH TEST CONDITIONS | - | | | | | | |
| ESTABLISH TEST FACILITIES | - | | | | | | |
| PREPARE SPECIMENS | - | | - | | | | |
| CONDUCT TESTS | - | | - | | | | |
| EVALUATE RESULTS | - | | - | | | | |
| ANALYZE DATA | - | | - | | | | |

Effect of Impurities in Coal-Derived Fuels on
Service Life of Boiler Tubes for Advanced Power
Cycle Applications

03-5

Coal/Materials and Components/Materials
Fireside Corrosion III

OBJECTIVES

Develop empirical heat flow/thermal conductivity models for multi-component refractory-lined gasifier vessel walls suitable for use in the design of commercial refractory linings for coal gasification vessels

PROJECT JUSTIFICATION

Heat flow calculations are usually based on thermal conductivity data measured on small laboratory samples. These calculations are only approximate and do not take into account the effect of metal anchors embedded in the refractory, the effect of cracks in the lining, and the effect of gas composition and pressure. Literature sources indicate significant changes when the pore system is filled with a high thermal conductivity gas such as hydrogen, especially when a high porosity insulating refractory is used, but the data, which are measured on small laboratory samples only are conflicting. No data are available on the effect of anchors or flaws in the lining. Thus, precise heat transfer calculations are not feasible, leading to the design of vessels with the possibility of too high or too low wall temperatures. These difficulties can be overcome by the use of steam or water jackets at the shell as is done in the Bi-Gas pilot plant. However, this is a costly solution. Failure of the steam or water cooling system may also cause premature shutdown. If the heat transfer through the refractory lining can be accurately calculated and the shell temperature predicted, external cooling can be eliminated and a safer operation of a coal gasification vessel can be obtained at significantly lower costs.

CONTRACT NO. E(49-18)-2210
CONTRACTOR Battelle Memorial Institute
ADDRESS 505 King Avenue
 Columbus, Ohio 43201

PRIN. INVEST. J. R. Schorr
BEGIN/END 1 Jun 1976 thru 30 May 1978

CONTRACT VALUE \$445,373

SPONSOR FER

DIRECTORATE Materials and Power Generation

TECH. PROJ. OFF. W. T. Bakker

CATEGORY/DETAIL Materials and Components/Materials

FUNDING (thousands of dollars)

| FY | ERDA | CONTRACTOR | TOTAL |
|----|------|------------|-------|
| 76 | 445 | — — | 445 |

ACTIVITY SCHEDULE

| ACTIVITY | FY 76 | TRANS | FY 77 | FY 78 | FY 79 | FY 80 | FY 81 |
|-------------------------------|-------|-------|-------|-------|-------|-------|-------|
| SURVEY LITERATURE | | | | | | | |
| DERIVE HEAT TRANSFER MODELS | | | | | | | |
| ACQUIRE FURNACES | | | | | | | |
| MEASURE HEAT TRANSFER | | | | | | | |
| CHARACTERIZE LINING MATERIALS | | | | | | | |
| CORRELATE DATA | | | | | | | |

PROJECT DESCRIPTION

This experimental program consists of the following six tasks:

- I. Survey appropriate literature on the thermal conductivity of refractories and on heat transfer through refractory linings. Prepare a critical evaluation report of existing literature.
- II. Derive mathematical models for heat transfer through single and multicomponent refractory walls with and without metal anchors, with and without flaws, provided sufficient literature data are available.
- III. Design, construct, or adapt heating furnaces capable of operating at temperatures up to 2300°F, suitable for heat transfer experiments through a refractory wall panel measuring at least 10 sq ft, using various gas compositions at atmospheric pressure. Preferably the furnaces should also be suitable for heat transfer studies at elevated pressures up to 1000 psi. However, since the construction of such furnaces may be prohibitively expensive, smaller furnaces may be preferred for high pressure studies.
- IV. Measure thermal gradients and heat transfer at thermal equilibrium conditions through a refractory panel in the furnace at 500, 1000, 1500, and 2000°F, using various lining designs and materials. The effects of the following variables are to be investigated on the basic lining design: anchor spacing, varying atmospheres and pressures, and flow size.
- V. Characterize the lining materials used and determine physical properties pertinent to the heat transfer tests: thermal conductivity, density, porosity, thermal expansion, hot and cold strength, and permeability. Perform nondestructive testing of lining before and after test to determine soundness of lining, if suitable methods are available. Determine physical properties of lining after test, at various points in the lining (unfired refractories only): LOI, state of hydration, density, porosity, and strength.
- VI. Correlate the data obtained with the theoretical heat transfer models obtained under Task II. If this is impractical, derive empirical heat flow models. These models should be usable for the design of commercial coal gasification vessels, preferably by means of a computer program.

OBJECTIVES

Reduce or eliminate mechanical deterioration of monolithic refractory linings of coal gasification process vessels

PROJECT JUSTIFICATION

Monolithic linings used in coal gasification pilot plants usually crack during dryout and initial heat-up. This is also common in other large process vessels lined with monolithic refractories, such as secondary reformers and catalytic crackers in the petrochemical industry, and soaking pits and rehear furnaces in the steel industry.

In addition to stresses and strains generated in the refractory lining during heat-up, additional stresses and strains will probably occur during the operation, especially during cyclic operations in which either the temperature, pressure, or atmosphere is changed. These stresses, together with corrosive and erosive environmental conditions, will probably lead to a gradual degradation of the refractory lining. The degree and rate of this deterioration process should be studied to produce a long-term service life in the refractory lining and to develop improved materials offering the promise of increased service life.

CONTRACT DATA

CONTRACT NO. E(49-18)-2218
CONTRACTOR The Babcock and Wilcox Co.
ADDRESS Lynchburg, Va. 24505

PRIN. INVEST. E. M. Anderson

BEGIN/END 29 Jun 1976 thru 28 Jun 1978

CONTRACT VALUE \$939,175

SPONSOR FER

DIRECTORATE Materials and Power Generation

TECH. PROJ. OFF. W. T. Bakker

CATEGORY/DETAIL Materials and Components/Materials

FUNDING
(thousands of dollars)

| FY | ERDA | CONTRACTOR | TOTAL |
|----|------|------------|-------|
| 76 | 800 | 107 | 907 |

PROJECT DESCRIPTION

This materials development project consists of the following eight tasks:

- I. Make a critical literature survey of the volume stability, mechanical properties, and chemical changes of monolithic refractories during curing, dryout, and heat-up, as related to crack formation in monolithic linings of large process vessels during heat-up.
- II. Derive mathematical models for stresses and strains occurring in 12-in. thick monolithic refractory linings of gasifier vessels of various sizes, when heated from room temperature to 2000°F, using materials properties available in the literature.
- III. Determine relevant mechanical properties of calcium aluminates- and phosphate-bonded monolithic refractories at temperatures in the 200 to 2000°F range.
- IV. Develop or modify monolithic refractory materials to improve their resistance to cracking during heat-up.
- V. Construct a cylindrical furnace having an I.D. of at least 24 in., a height of at least 48 in., and a lining thickness of 12 in. The furnace must have a temperature capability up to at least 2400°F with heat-up rates up to 300°F/hr and must be able to withstand pressures up to at least 250 psi. Instrument the furnace to measure temperature, stresses, and strains at various points in the lining and at the steel shell.
- VI. Measure stresses and strains (expansion/contraction) of the furnace lining as a function of temperature, heat-up rate, materials composition, and lining design. Variables to be investigated include effects of heat-up rate, lining materials, and design.
- VII. After the heat-up test, inspect the lining for physical damage (use NDT tests if applicable). If the lining is not too badly cracked, obtain samples from various points to determine key physical properties such as LOI, degree of hydration, density, porosity, and strength.
- VIII. Make the following correlations with the probability of crack formation in the refractory lining: physical properties of the materials; lining design (thickness, anchor spacing, and type); operating procedures (heat-up rate, atmospheric conditions, etc); and the effect of vessel size.

ACTIVITY SCHEDULE

| ACTIVITY | FY 76 | TRANS | FY 77 | FY 78 | FY 79 | FY 80 | FY 81 |
|------------------------------------|-------|-------|-------|-------|-------|-------|-------|
| SURVEY LITERATURE | | | | | | | |
| DEVELOP STRESS MODELS | | | | | | | |
| DETERMINE MECHANICAL PROPERTIES | | | | | | | |
| DEVELOP MONOLITHIC REFRACTORIES | | | | | | | |
| CONSTRUCT FURNACE | | | | | | | |
| MEASURE STRESS AT HIGH TEMPERATURE | | | | | | | |
| TEST LININGS AFTER HEATING | | | | | | | |
| CORRELATE DATA | | | | | | | |

Improvement of the Mechanical Reliability of
Monolithic Refractory Linings for Coal Gasification
Process Vessels

03-7

Coal/Materials and Components/Materials
Monolithic Refractory Linings

Conduct corrosion/erosion experiments on heat exchanger tube materials exposed to a high temperature (1300 to 1800°F) gaseous corrosion and particulate erosion environment in an atmospheric fluidized bed coal combustor (AFBCC) to establish engineering durability data as a function of materials properties.

PROJECT JUSTIFICATION

This project will gather realistic engineering data on the corrosion of heat exchanger materials in an atmospheric fluidized bed coal combustor for times up to 2000 hr. This testing will provide input data for future fluidized bed designs in the most direct and rapid way possible.

CONTRACT NO. E(49-18)-2325
CONTRACTOR Battelle Memorial Institute
ADDRESS 505 King Avenue
 Columbus, Ohio 43201
PRIN. INVEST. A. M. Hall
BEGIN/END 28 May 1976 thru 28 Aug 1977
CONTRACT VALUE \$766,637
SPONSOR FER/CCU
DIRECTORATE Materials and Power Generation
TECH. PROJ. OFF. S. J. Dapkunas
CATEGORY/DETAIL Materials and Components/Materials

FUNDING (thousands of dollars)

| FY | ERDA | CONTRACTOR | TOTAL |
|----|------|------------|-------|
| 76 | 580 | -- | 580 |

This materials evaluation project consists of high temperature corrosion/erosion experiments on candidate heat exchanger and superheater tube materials exposed to gaseous corrosion and particulate erosion in and above the bed of an AFBCC containing limestone as a sulfur oxide sorbent.

Include 9 Cr-1.5 Mo, 304 stainless steel, HK-40 (310), Inconel 671, and X-40, as well as either the 304 stainless or the HK-40 with a protective coating, as materials for in- and above-bed testing. Assemble materials into air-cooled probes consisting of 18 cylindrical specimens, each with thermocouples located in the probe so that individual specimen surface temperatures can be determined. Air-cool the specimen probes to maintain a temperature gradient from 1300 to 1800°F over the length of the probes.

Initiate a single 2000 hr run in the AFBCC. Insert two corrosion probes in the bed and two in the space above the bed at the start-up of the run. After 10 hr of operation, remove one probe at each position, refill them with specimens, and reinsert them into the combustor for a period of 500 hr. Allow the other two probes to remain in the AFBCC for 1500 hr or longer.

Analyze each lot of coal and determine the composition of the ash and sorbents. Take gas samples from above the fluidized bed and correlate these data, where possible, with the chemical data gathered above and with the operating conditions. Take samples at the start of the 10-hr run to characterize the exposure condition at the probe locations. Make additional analyses at the time the first probes are removed (10 hr), repeat the analysis at the end of the 500-hr exposure, and at the conclusion of the 1500-hr minimum exposure. Make in-bed measurements of gas particle velocity near the specimen probes. Maintain a complete characterization (chemical and microstructural) of the test materials. Assess material performance, make comparative metallographic examinations of each material evaluated, and obtain hardness and bend test data.

ACTIVITY SCHEDULE

| ACTIVITY | FY 76 | TRANS | FY 77 | FY 78 | FY 79 | FY 80 | FY 81 |
|---------------------------|-------|-------|-------|-------|-------|-------|-------|
| ESTABLISH TEST FACILITIES | | ===== | | | | | |
| PREPARE SPECIMENS | | ===== | | | | | |
| CONDUCT TESTS | | | ===== | | | | |
| EVALUATE RESULTS | | | ===== | | | | |

OBJECTIVES

Provide reliable, long-term engineering data on the corrosion/erosion deterioration of:

Gas turbine materials exposed to the exhaust gas from a pressurized fluidized bed coal combustor (PFBCC)

PROJECT JUSTIFICATION

This program will gather data, now lacking, on the corrosion/erosion behavior of materials used in gas turbines run off a pressurized fluidized bed coal combustor. The technique will yield reliable data in a most straightforward, rapid manner.

CONTRACT DATA

CONTRACT NO. E(49-18)-2326
 CONTRACTOR General Electric Company
 ADDRESS 1 River Road
 Schenectady, N.Y. 12345
 PRIN. INVEST. R. G. Frank
 BEGIN/END 1 July 1976 thru 30 June 1978
 CONTRACT VALUE \$240,035
 SPONSOR FER/CCU
 DIRECTORATE Materials and Power Generation
 TECH. PROJ. OFF. S. J. Dapkunas
 CATEGORY/DETAIL Materials and Components/Materials

FUNDING
 (thousands of dollars)

| FY | ERDA | CONTRACTOR | TOTAL |
|----|------|------------|-------|
| 76 | 100 | -- | 100 |

PROJECT DESCRIPTION

This materials evaluation project consists of the following three tasks:

- I. Conduct small burner tests using synthesized atmospheres and/or deposits representative of those in gases exhausting from a pressurized fluidized bed coal combustor (PFBCC). Perform the tests at a specimen temperature of 1600°F, using a maximum of four candidate turbine bucket and vane materials selected on the basis of existing corrosion rates in fluidized bed combustor atmospheres. Obtain and document microstructures and chemistries of these materials. Use exposure times up to 7000 hr, removing and evaluating specimens at intermittent periods with the objective of developing a relationship between time and penetration. Evaluate specimens metallographically for average metal recession and maximum penetration due to corrosion. Where appropriate, conduct chemical analysis of deposits and metallographic analysis of surface morphology on selected samples.
- II. Fabricate specimens of the same materials tested in Task I for testing in the Exxon miniplant for times up to 1100 hr. Obtain and document microstructures and chemistries of these materials. Metallographically evaluate the specimens after exposure for average metal recession and maximum penetration due to erosion and corrosion. Where appropriate, conduct chemical analysis of deposits and metallographic analysis of surface morphology on selected samples. Compare erosion/corrosion behavior of exposed samples with alloy composition.
- III. Compare chemistry of deposits and morphology of surfaces of specimens exposed in Task I and II. Based on Task II corrosion/erosion data, estimate the lives of parts for several assumed contaminant levels and operating temperatures and pressures; use proprietary models and computer programs to generate these estimates.

ACTIVITY SCHEDULE

| ACTIVITY | FY 76 | TRANS | FY 77 | FY 78 | FY 79 | FY 80 | FY 81 |
|---|-------|-------|-------|-------|-------|-------|-------|
| CONDUCT SMALL BURNER CORROSION/EROSION TESTS | | | ===== | | | | |
| FABRICATE AND EVALUATE SPECIMENS FOR MINIPANT TESTS | | | ===== | | | | |
| COMPARE SAMPLES FROM BOTH TESTS | | | | ===== | | | |

Determine quantitative corrosion/erosion data on selected heat exchanger materials exposed in the Exxon pressurized fluidized bed coal combustor (PFBCC) for times up to 1100 hr.

PROJECT JUSTIFICATION

This effort will generate reliable, quantitative data on the corrosion/erosion of selected heat exchanger and turbine materials by laboratory screening followed by exposure in a PFBCC. Realistic corrosion/erosion engineering data will be gathered in the Exxon Research and Engineering PFBCC miniplant over an accumulated operating time of 1100 hours.

This program complements atmospheric fluidized bed coal combustor programs at the Battelle Memorial Institute and the General Electric Company. One benefit to be gained by this effort will be insight into the necessity for testing to ascertain corrosion/erosion under pressurized conditions. A significant advantage that this Westinghouse program offers is the insight to be gained on the behavior of welded materials and the effects of steam corrosion/erosion on the interior of superheater tubes.

CONTRACT NO. E(49-18)-2327
 CONTRACTOR Westinghouse Electric Corporation
 ADDRESS 1310 Beulah Road
 Pittsburgh, Penn. 15235
 PRIN. INVEST. D. L. Keairns
 BEGIN/END 1 Jul 1976 thru 30 Jun 1978
 CONTRACT VALUE \$244,644
 SPONSOR FER/CCU
 DIRECTORATE Materials and Power Generation
 TECH. PROJ. OFF. S. J. Dapkunas
 CATEGORY/DETAIL Materials and Components/Materials

FUNDING (thousands of dollars)

| FY | ERDA | CONTRACTOR | TOTAL |
|----|------|------------|-------|
| 76 | 100 | -- | 100 |

This project to evaluate materials for corrosion/erosion consists of tasks to:

- I. Select coal and sorbent feedstocks for tests in the Exxon miniplant. Evaluate HS-188, Incoloy 800, Hastelloy X, and type 304 stainless steel, as a minimum, for above-bed superheaters. Evaluate 2.25 Cr-1 Mo steel, 9 Cr-1.5 Mo steel, Hastelloy X, and HS-188 for in-bed heat exchangers. Include welded joints of both types of materials. Use temperatures of 1050°F for the Cr-Mo steels, 1200°F for the Incoloy 800 and 304 SS, 1400°F for the Hastelloy X and Incoloy 800, and 1600°F for the HS-188 and Hastelloy X alloys. Expose these alloys in the Exxon miniplant for 1100 hr using selected feedstocks. Remove test specimens for metallurgical examination at 100, 500, and 1000 hr and at other times if required by corrosion/erosion deterioration. Analyze coals and sorbents for various chemical elements, including alkali and alkaline earth metals. Characterize all alloys chemically and metallurgically. Evaluate tested materials (including welds and regions in contact with steam) for extent of material recession due to erosion, together with extent of corrosion deterioration as measured by surface loss and corrosive penetration. Analyze the chemical and physical nature of surface deposits and scale on specimens.
- II. Run simulation tests in a pressurized fluidized bed environment at the same temperature-alloy combinations as in Task I, and at a pressure of 10 atm with the atmospheres synthesized on the basis of the Exxon miniplant results. Minimum test time is to be 100 hr. Characterize exposed specimens in terms of surface scale, deposits, and corrosive penetration. Correlate the results with the corrosion morphologies obtained in the Exxon miniplant tests.

Throughout Tasks I and II, attempt to provide test data to permit correlations of specimen corrosion/erosion with: fluidized bed miniplant feedstock characterization and operating conditions, alloy composition, in-plant above-bed gas characterization, specimen temperature, and times of exposure up to 1000 hr. Correlate characterized deposits on specimens with operating conditions.

ACTIVITY SCHEDULE

| ACTIVITY | FY 76 | TRANS | FY 77 | FY 78 | FY 79 | FY 80 | FY 81 |
|-----------------------------------|-------|-------|-------|-------|-------|-------|-------|
| PREPARE SPECIMENS | | ===== | | | | | |
| PERFORM CORROSION/EROSION TESTING | | ===== | | | | | |
| RUN SIMULATION TESTS | | ===== | | | | | |
| CONDUCT ANALYSES | | ===== | | | | | |

Operate a pressurized, fluidized bed coal combustor for 1100 hr to provide a test site for exposure of heat exchanger and gas turbine materials by Westinghouse and General Electric, respectively

PROJECT JUSTIFICATION

The development of fluidized bed coal combustors has necessitated a knowledge of corrosion/erosion rates for both heat exchanger and gas turbine materials. The Fireside Corrosion Program, Task II (Evaluation of Heat Exchanger and Turbine Materials for Use in a Coal-Fired Fluidized Bed Combustion Environment) was initiated to gather this data.

Westinghouse Electric Corporation will evaluate heat exchanger materials for in- and above-bed applications in a pressurized, fluidized bed coal combustor (PFBCC) and the General Electric Company will evaluate materials used in a gas turbine operating in the exhaust gases of a PFBCC. Both companies will expose their materials in the PFBCC operated by Exxon for the EPA, performing their tests during the same period of PFBCC operation to reduce test costs.

CONTRACT NO. E(49-18)-2452
 CONTRACTOR Exxon Research & Engineering Co.
 ADDRESS P.O. Box 8
 Linden, N.J. 07036
 PRIN. INVEST. R. Bertrand
 BEGIN/END 1 Jul 1976 thru 31 Dec 1977
 CONTRACT VALUE \$871,124
 SPONSOR FER/CCU
 DIRECTORATE Materials and Power Generation
 TECH. PROJ. OFF. S. J. Dapkunas
 CATEGORY/DETAIL Materials and Components/Materials

FUNDING (thousands of dollars)

| FY | ERDA | CONTRACTOR | TOTAL |
|----|------|------------|-------|
| 76 | 720 | -- | 720 |

This project consists of the following tasks:

- I. Design modifications to the existing pressurized fluidized bed facility to accept the turbine test passage supplied by General Electric and the in-bed and above-bed heat exchanger probes to be supplied by Westinghouse.
- II. Fabricate the modifications designed in Task I and install the General Electric turbine test passage together with supporting utilities required for its operation and the Westinghouse heat exchanger probes with their required supporting utilities.
- III. Conduct tests according to the following schedule:
 - a. 100-hr shakedown test to assess the performance of heat exchanger probes and turbine test passage in conjunction with the granular bed gas filter. This test will establish the effect of test equipment on bed operation and provide an opportunity for General Electric and Westinghouse to evaluate materials after a short test run. This test is to be conducted during one work week.
 - b. 1000-hr test to expose heat exchanger and gas turbine materials supplied by Westinghouse and General Electric, respectively, for up to 1000 hr. This test will be interrupted at three points (at approximately 200, 500, and 700 hr) to allow removal, evaluation, and reinstallation of specimens.

During both these tests Exxon will monitor and control the temperature of heat exchanger probes and materials in the gas turbine test passage, maintaining turbine passage inlet gas in the 1400 to 1650°F range. It will provide a 100 point data logger to continuously monitor these specimen temperatures, monitor the composition of effluent gas from the bed and gas entering the turbine test passage during all runs, and monitor the particle size distribution and particle loading in gas above the bed and in gas entering the turbine test passage. It will maintain a profile of bed conditions, including temperatures, pressure, and velocity for each run.

ACTIVITY SCHEDULE

| ACTIVITY | FY 76 | TRANS | FY 77 | FY 78 | FY 79 | FY 80 | FY 81 |
|-------------------------------|-------|-------|-------|-------|-------|-------|-------|
| DESIGN FACILITY MODIFICATIONS | | | | | | | |
| INSTALL EQUIPMENT | | | | | | | |
| RUN 100-HR SHAKEDOWN TEST | | | | | | | |
| CONDUCT 1000-HR TEST | | | | | | | |

Operate EPA Pressurized Fluidized Bed Combustor

03-11

Coal/Materials and Components/Materials
Fireside Corrosion II

Make services of ceramic specialists at Amoco available to assist in the design of refractory linings for coal gasification plants, for failure analysis, and for evaluation of work performed by ERDA contractors on ceramic materials

PROJECT JUSTIFICATION

Operating conditions in coal gasifier vessels have much in common with those in some petrochemical processes, especially in secondary reformers. Therefore, refractory lining practices used in these reformers are being used in coal gasification pilot plants. Lining practices within the petrochemical industry vary considerably depending on the size and operating conditions of the process equipment. Thus, a single, codified, and published lining practice similar to the ASME boiler and pressure vessel codes is not available. Instead, each lining must be custom designed, based on the requirements and past experience with similar designs. Thus, the success of a given design depends greatly on the skill and experience of the design engineer.

Within the petrochemical and refractories industry, Amoco personnel and especially Dr. Cowley are recognized as leading experts on the design and installation of refractories in petrochemical process vessels. In the past, ERDA has used the services of Dr. Cowley on an ad hoc basis, and it is beneficial to have assistance of Amoco specialists available on a limited basis to provide ERDA with the best possible refractory advice from the petrochemical industry.

CONTRACT NO. Purchase Order 76-4
CONTRACTOR Standard Oil Company (Indiana)
ADDRESS P.O. Box 400
 Napierville, Ill. 60540
PRIN. INVEST. M. S. Crowley
BEGIN/END 16 Oct 1975 thru 15 Oct 1976
CONTRACT VALUE \$4,500
SPONSOR FER
DIRECTORATE Materials and Power Generation
TECH. PROJ. OFF. W. T. Bakker
CATEGORY/DETAIL Materials and Components/Materials

FUNDING (thousands of dollars)

| FY | ERDA | CONTRACTOR | TOTAL |
|----|------|------------|-------|
| 76 | 5 | -- | 5 |

This purchase agreement provides for the following tasks:

- I. Review designs of refractory linings for coal conversion process vessels.
- II. Assist ERDA and its contractors with the analysis of refractory linings after service.
- III. Evaluate the work of contractors engaged in projects on ceramic materials.

ACTIVITY SCHEDULE

| ACTIVITY | FY 76 | TRANS | FY 77 | FY 78 | FY 79 | FY 80 | FY 81 |
|--------------------------|-------|-------|-------|-------|-------|-------|-------|
| REVIEW LINING DESIGNS | | | | | | | |
| ASSIST IN ANALYSIS | | | | | | | |
| EVALUATE CONTRACTOR WORK | | | | | | | |

- Evaluate refractory materials for the Bi-Gas process
- Develop erosion-resistant ceramic coatings for metal parts
- Develop NDT methods for coal gasification plant components
- Conduct failure analyses of failed pilot plant components

PROJECT JUSTIFICATION

Project Justification

A study by the Metals Properties Council in 1972 for the Office of Coal Research (OCR) indicated that corrosion problems could be expected with both metallic and ceramic materials for all of the coal gasification processes being considered at that time. Work was initiated by OCR and expanded by ERDA to develop materials that could withstand the corrosive coal gasifier environments, thereby improving the economic feasibility of commercial coal gasification systems.

This project will complement other materials research programs sponsored by the ERDA Division of Fossil Energy Research to provide materials support, engineering data, and quality assurance methods for coal gasification processes. Data resulting from this project should increase the reliability and decrease the cost of coal gasification plants.

CONTRACT NO. ANL Project 7106
CONTRACTOR Argonne National Laboratory
ADDRESS 9700 So. Cass Avenue
 Argonne, Ill. 60439
PRIN. INVEST. R. W. Weeks
BEGIN/END 1 Oct 1974 thru 30 Sep 1977
CONTRACT VALUE \$2,114,000
SPONSOR FER
DIRECTORATE Materials and Power Generation
TECH. PROJ. OFF. W. T. Bakker
CATEGORY/DETAIL Materials and Components/Materials

FUNDING (thousands of dollars)

| FY | ERDA | CONTRACTOR | TOTAL |
|----|------|------------|-------|
| 75 | 560 | -- | 560 |
| 76 | 604 | -- | 604 |
| TQ | 200 | -- | 200 |

This continuation of the ANL materials technology program consists of the following six tasks:

- I. Initial static screening indicated that only about 4 of the 24 refractories tested appear to have the potential to survive the corrosive environment of the slagging gasifier. Of these, the Cr_2O_3 solid solution bonds and the fused-glassy bonds survived best. Using the results of these screening tests, choose refractories for testing under dynamic corrosive/erosive conditions in the newly completed test rig. Conduct tests of up to 1000 hr duration that will closely simulate the conditions in the Bi-Gas gasifier.
- II. Evaluate ceramic coatings for coal-conversion plants. Erosive screening tests have been performed under subcontract by the Solar Corporation on a number of commercially supplied coatings (oxides, carbides, nitrides, and borides) and substrates (carbon and stainless steels and high-nickel alloys). Prepare the remaining samples to be tested, including boride and nitride coatings applied by the plasma spray technique. After completion of the erosion testing, arrange to place coated test elements, such as piping elbows, in pilot plant service for evaluation.
- III. Conduct NDT of pilot plant components. Determine appropriate NDT methods to be used on various material combinations encountered in pilot plants under a wide variety of operating conditions. As pilot plant data become available, develop the capability to interpret these data in terms of the operational problems that exist in these plants.
- IV. Develop correlations that depict the corrosion-erosion behavior of materials as a function of process conditions to permit extrapolations and predictions of long-term performance of specific components.
- V. Conduct a number of critical erosion-wear experiments, including testing of materials that have undergone corrosive degradation, for validation of the analytical models and for effective use of the models to predict metal loss under erosive-corrosive conditions in coal gasification plants.
- VI. Continue to perform failure analyses, as required, for pilot plants. The extent of a given analysis will depend on the importance of the component to the pilot plant operation and the difficulty in determining the cause of the failure.

ACTIVITY SCHEDULE

| ACTIVITY | FY 76 | TRANS | FY 77 | FY 78 | FY 79 | FY 80 | FY 81 |
|--|-------|-------|-------|-------|-------|-------|-------|
| EVALUATE CERAMIC REFRACTORIES | ===== | | | | | | |
| EVALUATE CERAMIC COATINGS | ===== | | | | | | |
| DEVELOP NDT TECHNIQUES | ===== | | | | | | |
| DEVELOP CORROSION-EROSION CORRELATIONS | ===== | | | | | | |
| CONDUCT EROSION-WEAR EXPERIMENTS | ===== | | | | | | |
| PERFORM FAILURE ANALYSIS | ===== | | | | | | |

- Collect existing data and practices specifically related to coal liquefaction and gasification processes.
- Evaluate data and prepare report on materials selection for pressure vessels and piping for coal conversion plants based on design criteria.
- Recommend extensions to existing codes and areas where additional data should be developed to improve design capability.

PROJECT JUSTIFICATION

The need for a coal conversion pressure vessel and piping technology assessment document becomes clear when specific design criteria guidelines are sought. The ASME "Boiler and Pressure Vessel Codes" do not specifically cover the load, temperature, and environmental conditions associated with the coal conversion processes. Therefore, the ASME codes have limited applicability for these processes. This project will result in the collection of the many fragmented, but extremely important, bits of documented information in this area, leading to a credible approach for the formulation of design criteria pertaining to coal conversion pressure vessel and piping technology.

CONTRACT NO. ORNL Project 7145

CONTRACTOR Oak Ridge National Laboratory
ADDRESS P.O. Box X
Oak Ridge, Tenn. 37830

PRIN. INVEST. D. A. Canonico

BEGIN/END 1 Jan 1976 thru 30 Jun 1976

CONTRACT VALUE \$95,000

SPONSOR FER

DIRECTORATE Materials and Power Generation

TECH. PROJ. OFF. T. B. Cox

CATEGORY/DETAIL Materials and Components/Materials

FUNDING (thousands of dollars)

| FY | ERDA | CONTRACTOR | TOTAL |
|----|------|------------|-------|
| 76 | 95 | — — | 95 |

This technology assessment project consists of the following five tasks:

- I. Collect, document, and assess those environmental and boundary conditions critical to design criteria of coal conversion system components, including data on: gaseous and fluid stream composition, static and dynamic pressure, temperatures, and localized loading.
- II. Assess and compile a listing of those construction materials considered for and/or used in coal conversion processes and determine what, if any, derating factors were used by the designer to incorporate known or unknown facts, such as joints, welds, and interfaces. In addition, assess the efficacy with which the designer used mechanical and physical properties; susceptibility to hydrogen embrittlement, stress corrosion, erosion, carburization, and oxidation; weldability; experience of fabricators with chosen materials; and fabrication costs.
- III. Assess size limitations placed on pressure vessels and piping. In addition, investigate and report on the following topics related to structural integrity: effects of probable flaws and effects of geometrical design change on failure modes and stress distributions.
- IV. Review and assess quality assurance techniques and systems applicable to pressure vessel and piping systems.
- V. Review and assess surveillance procedures now being used and applicable to pressure vessel and piping systems of coal conversion processes. These procedures apply to inspection of components during shut-down periods, continuous in-service monitoring of material performance, and pre- and post-operational pressure testing for system integrity.

ACTIVITY SCHEDULE

| ACTIVITY | FY 76 | TRANS | FY 77 | FY 78 | FY 79 | FY 80 | FY 81 |
|--|-------|-------|-------|-------|-------|-------|-------|
| COLLECT AND ASSESS DATA ON BOUNDARY CONDITIONS | _____ | | | | | | |
| ASSESS CONSTRUCTION MATERIALS | _____ | | | | | | |
| INVESTIGATE FLAW AND GEOMETRY EFFECTS | _____ | | | | | | |
| ASSESS QUALITY ASSURANCE TECHNIQUES | _____ | | | | | | |
| REVIEW SURVEILLANCE PROCEDURES | _____ | | | | | | |

Obtain pertinent information with respect to liquefaction processes, based on analyses of failures, to prevent recurrences and thus contribute to the reliability of liquefaction plants

PROJECT JUSTIFICATION

Materials and components of coal conversion systems are subjected to various debilitating conditions, most of them occurring at high temperature: oxidation, corrosion (from sulfur and other elements in the coal), and erosion (mainly from the relatively high velocity, hot streams carrying particles from the coal). In addition, at relatively high temperature, materials lose a portion of their strength, as determined by simple uniaxial tests, although the parts are generally under some complex multistress system. To achieve better performance, analysis of these failures is obligatory.

CONTRACT NO. ORNL Project 7146
CONTRACTOR Oak Ridge National Laboratory
ADDRESS P.O. Box X
 Oak Ridge, Tenn. 37830

PRIN. INVEST. D. A. Canonico
BEGIN/END 1 Jan 1976 thru 30 Sep 1976

CONTRACT VALUE \$100,000

SPONSOR FER

DIRECTORATE Materials and Power Generation

TECH. PROJ. OFF. W. T. Bakker

CATEGORY/DETAIL Materials and Components/Materials

FUNDING (thousands of dollars)

| FY | ERDA | CONTRACTOR | TOTAL |
|----|------|------------|-------|
| 76 | 100 | -- | 100 |

Oak Ridge National Laboratory will analyze materials and component failures in coal liquefaction plants and advise on solutions for the failures, as requested by ERDA.

ACTIVITY SCHEDULE

| ACTIVITY | FY 76 | TRANS | FY 77 | FY 78 | FY 79 | FY 80 | FY 81 |
|---|-------|-------|-------|-------|-------|-------|-------|
| ANALYZE MATERIAL AND COMPONENT FAILURES | | | | | | | |

- Investigate material and component failures in ERDA's fossil energy process development units, pilot plants, and demonstration plants.
- Recommend improvements in materials and components, based on failure analysis.

PROJECT JUSTIFICATION

Fossil Energy Research has organized a materials and components failure analysis system covering all fossil energy process development units, pilot plants, and demonstration plants to provide reliable and durable materials for coal conversion processes. Contracts to perform failure analysis are presently in force with laboratories located in the eastern U.S. where most of the experimental plants are located. If a failure analysis system is to be effective, its response to failure occurrences must be quick.

At present this cannot be accomplished for pilot plants located in the western U.S. It is, therefore, desirable to initiate a limited failure analysis program in a western laboratory. Since FER already has a materials program at Sandia Laboratories in California, it is best to have this program at the same location. Then, materials people from the concurrent program can be used for failure analysis if and when a failure occurs.

CONTRACT NO. SL Project 7xxx
 CONTRACTOR Sandia Laboratories
 ADDRESS Livermore, Calif. 94550

PRIN. INVEST. A. J. West
 BEGIN/END 1 Jan 1976 thru 30 Sep 1976
 CONTRACT VALUE \$25,000/yr
 SPONSOR FER
 DIRECTORATE Materials and Power Generation
 TECH. PROJ. OFF. W. T. Bakker
 CATEGORY/DETAIL Materials and Components/Materials
FUNDING
 (thousands of dollars)

| FY | ERDA | CONTRACTOR | TOTAL |
|----|------|------------|-------|
| 76 | 11 | -- | 11 |
| TQ | 14 | -- | 14 |

This failure analysis program consists of three tasks to:

- I. Investigate material and component failures in ERDA/FE installations by visual inspection on the site, laboratory investigation of the failed materials or components, and by other methods considered necessary.
- II. Recommend improved materials and components or design changes, aimed at preventing further failures and improving the system reliability.
- III. Disseminate the knowledge acquired for use in other applicable fossil energy installations.

ACTIVITY SCHEDULE

| ACTIVITY | FY 76 | TRANS | FY 77 | FY 78 | FY 79 | FY 80 | FY 81 |
|---|-------|-------|-------|-------|-------|-------|-------|
| PERFORM FAILURE ANALYSIS WHEN REQUESTED BY ERDA | | | | | | | |
| INVESTIGATE FAILURES | | | | | | | |
| RECOMMEND IMPROVED MATERIALS AND COMPONENTS | | | | | | | |
| DISSEMINATE INFORMATION | | | | | | | |

- Analyze processes by which materials corrode, wear, or break in coal conversion systems
- Provide data on construction materials for coal conversion processes

PROJECT JUSTIFICATION

The high-temperature, high-pressure coal gasification processes currently being developed require construction materials that can withstand more rigorous conditions than those previously encountered. As these new coal gasification technologies are developed, new test methods and data must be produced to ensure the reliability and durability of materials subjected to these new environments. The benefits from improved critical part life or avoidance of unexpected material failures in coal gasification plants would be of considerable economic importance. In some cases, these considerations may even determine whether a particular process is viable. Pilot plant experience indicates that tests are needed for corrosion, fracture, and wear of both metal and ceramic parts.

Consequently, the ERDA and the NBS have initiated a program to develop improved test procedures (especially short-term tests) for materials that are candidates for use in coal gasification plants. Emphasis in the initial program is on tests for susceptibility of materials to erosive wear and corrosion-aided fracture in coal gasification environments.

CONTRACT NO. E(49-18)-1749
CONTRACTOR National Bureau of Standards
ADDRESS Gaithersburg, Md. 20234

PRIN. INVEST. S. J. Schneider
BEGIN/END 1 Jul 1974 thru 30 Sep 1977
CONTRACT VALUE \$875,000
SPONSOR FER
DIRECTORATE Materials and Power Generation
TECH. PROJ. OFF. W. T. Bakker
CATEGORY/DETAIL Materials and Components/Materials

FUNDING (thousands of dollars)

| FY | ERDA | CONTRACTOR | TOTAL |
|----|------|------------|-------|
| 75 | 875 | 875 | 1,750 |

Test methods recently developed or currently under investigation in corrosion-aided fracture are being expanded to test materials under coal gasification conditions. Toward that goal, this study consists of the following tasks:

- I. Build equipment to apply constant-strain-rate test techniques to determine metal susceptibility to stress corrosion cracking in coal gasification atmospheres.
- II. Develop techniques to permit improved screening of materials for use in coal gasification plants.
- III. Develop a test apparatus that will produce erosive wear at high temperatures and pressures similar to coal gasification conditions.
- IV. Conduct tests with this equipment to measure erosive wear in a controlled environment.
- V. Compare wear samples exposed in operating coal gasification facilities with Task IV test samples.

ACTIVITY SCHEDULE

| ACTIVITY | FY 76 | TRANS | FY 77 | FY 78 | FY 79 | FY 80 | FY 81 |
|--|-------|-------|-------|-------|-------|-------|-------|
| STUDY CHEMICAL DEGRADATION AND DEVELOP TESTS | | | | | | | |
| DEVELOP EROSION WEAR TEST APPARATUS | | | | | | | |
| CONDUCT EROSION WEAR TESTING | | | | | | | |
| DEVELOP STRESS CORROSION TEST | | | | | | | |

OBJECTIVES

Develop improved valve materials having the wear, abrasion, erosion, and shock resistance required to operate in coal gasification systems

PROJECT JUSTIFICATION

There is no simple method to feed dry coal to the high pressure gasifiers used for coal gasification, but lock hoppers are often used. Although the high power normally required to operate lock hoppers can be reduced by using two hoppers and venting from one to the other, several percent of the synthesis gas is still required for the pressurizing energy without any leakage from the isolating valves. If the valves leak, as is still the case, the energy requirements become excessive.

A similar problem exists with the char removal valves that now operate at 600°F. Continuous char withdrawal would be advantageous if the hot char and associated steam could be throttled through a small valve. This could not be designed into the PERC Synthane plant because current valve materials cannot withstand the high temperature abrasion. However, such a system would be highly desirable because the hot char could then be used to make steam.

Work being done at MERC and PERC has shown that a hard seat can be made to work well in a ball valve, probably because of the high pressures involved. Therefore, existing valves can be improved and new valves can be designed using exceptionally hard and wear-resistant materials.

This program with the Bureau of Mines, which uses this approach, is a continuation of work started in FY 1975 with Bureau of Mines funds to provide metallurgical support for the Synthane pilot plant.

CONTRACT NO. E(49-18)-2219-1
CONTRACTOR Bureau of Mines
ADDRESS Metallurgy Research Centers at Albany, Ore.; Rolla, Mo.; and College Park, Md.
PRIN. INVEST. Wood(A)/Stephenson(R)/Needham(CP)
BEGIN/END 1 Jul 1975 thru 30 Jun 1976
CONTRACT VALUE \$1,250,000 (for -2219-1 and -2)
SPONSOR FER
DIRECTORATE Materials and Power Generation
TECH. PROJ. OFF. W. T. Bakker
CATEGORY/DETAIL Materials and Components/Materials

FUNDING
(thousands of dollars)

| FY | ERDA | CONTRACTOR | TOTAL |
|----|------|------------|-------|
| 76 | 1250 | 400 | 1650 |

PROJECT DESCRIPTION

This materials development project consists of the following tasks:

- I. Continue to develop and evaluate hard, durable, wear-resistant valve materials, such as Ni-Cr bonded WC, sintered materials containing carbonitrides, and cobalt-base Stellite-like alloys.
- II. Improve and expand a previously established facility for wear testing of materials, especially at elevated temperatures. Continue wear tests to evaluate developmental materials and to screen candidate materials for making valve hardware.
- III. Complete the design and construction of a large molten-salt coating cell capable of coating valve seats and balls up to 14 in. in diameter with TiB₂. Develop engineering capabilities for coating large valve components with TiB₂. Study the physical properties of TiB₂ coatings produced under various conditions, with particular emphasis on the coating substrate adherence characteristics, crystal orientation, and corrosion and erosion resistance. Produce coated valve components with TiB₂ for in situ testing in the producer gas facility at MERC.
- IV. Continue to develop laboratory techniques for applying, by chemical vapor deposition (CVD), wear-resistant coatings of selected materials to ferrous alloy surfaces. Determine hardness, strength of bonding to substrate, wear-resistance, and uniformity and consistency of CVD coatings. Enlarge scale of CVD laboratory apparatus to facilitate coating larger hardware and the rapid transfer of technology to a commercial scale. Prepare tungsten, titanium nitride, and titanium carbonitride coatings on large ball valve seats by the CVD method.
- V. Produce or have produced critical valve parts of one or more of the most promising material and fabrication techniques for in-plant testing in one of the coal gasification pilot plants.

ACTIVITY SCHEDULE

| ACTIVITY | FY 76 | TRANS | FY 77 | FY 78 | FY 79 | FY 80 | FY 81 |
|--|-------|-------|-------|-------|-------|-------|-------|
| DEVELOP IMPROVED VALVE MATERIALS | | | | | | | |
| IMPROVE FACILITY FOR WEAR-TESTING AND CONTINUE TESTING | | | | | | | |
| DEVELOP TiB ₂ CAPABILITY | | | | | | | |
| DEVELOP CVD CAPABILITY | | | | | | | |
| MAKE AND TEST HARDWARE | | | | | | | |

- Determine corrosive effect of the principal constituent of the coal gasification atmosphere on selected refractories
- Determine if centrifugal casting is a feasible technique to coat large diameter lines in coal gasification vessels
- Eliminate spalling and cracking in ceramic liners

PROJECT JUSTIFICATION

Refractory linings used in coal gasification vessels have generally been patterned after those used in secondary reformers in plants making ammonia according to the steam-methane reforming process. Conditions in this process are somewhat similar to those encountered in coal-gasification vessels since a steam reforming plant is basically a coal gasification plant in reverse. However, there are significant differences, including the presence of considerably more steam in the coal gasification vessels and more CO and less H₂. In most gasification processes, operating pressures will be appreciably higher, but temperatures will be lower. In addition, the coal gasification atmosphere will contain various minor impurities that may corrode refractories, such as alkali vapors and H₂S.

Coal gasification pilot plants have been lined with refractories used in secondary reformers because insufficient data exist on alternative refractories more suitable for the gasifiers. This research program will assist in establishing proper refractory practices for large commercial gasifiers, thereby providing highly reliable, long-lived, lower cost refractories.

CONTRACT NO. E(49-18)-2219-2
CONTRACTOR Bureau of Mines
ADDRESS Tuscaloosa Metallurgy Research Center
 Tuscaloosa, Al.
PRIN. INVEST. H. Heystek/L. Sadler
BEGIN/END 1 Jul 1975 thru 30 Jun 1976
CONTRACT VALUE \$1,250,000 (for 2219-1 and -2)
SPONSOR FER
DIRECTORATE Materials and Power Generation
TECH. PROJ. OFF. W. T. Bakker
CATEGORY/DETAIL Materials and Components/Materials

FUNDING (thousands of dollars)

| FY | ERDA | CONTRACTOR | TOTAL |
|----|------|------------|-------|
| 76 | 1250 | 400 | 1650 |

This materials development project consists of the following tasks:

- I. Conduct a critical literature survey of the reactions between various gaseous atmospheres and refractories, especially those containing H₂, CO, H₂O, H₂S, and alkali metal vapors, and of the usage of refractories in high temperature furnaces containing such atmospheres.
- II. Assemble, check out, and debug a high temperature, high pressure furnace together with its auxiliary gas metering and monitoring equipment to ensure trouble-free test runs up to 1000 hr.
- III. Prepare suitable samples of the various refractories for exposure to simulated gasifier atmospheres in the test furnace.
- IV. Determine key properties of the refractories to be tested to establish baseline data against which the corrosive effects of the gasification atmosphere can be measured.
- V. Conduct preliminary screening tests for 100 to 150 hr at 1000 psi pressure and 1000° C using the MPC simulated coal gasification atmosphere, steam, H₂, CO, and CO/H₂/steam mixtures. Measure the same properties, as established in Task IV, for the samples after exposure. Determine structural and compositional changes if significant changes in physical properties are found.
- VI. Correlate and critically review all experimental data to establish potential failure mechanisms for the various classes of refractories tested, to predict useful service life, and to provide leads for the development of improved refractories.
- VII. Develop centrifugal casting technology for applying refractory concretes to large diameter pipes used as transfer lines in coal conversion processes.
- VIII. Continue the investigation of techniques for incorporating randomly oriented chopped fibers and wires for resistance to spalling and for arresting cracks, including fiber and wire reinforcement for both centrifugally cast and gunnited material.

ACTIVITY SCHEDULE

| ACTIVITY | FY 76 | TRANS | FY 77 | FY 78 | FY 79 | FY 80 | FY 81 |
|---|-------|-------|-------|-------|-------|-------|-------|
| TEST REFRACTORIES IN COAL GASIFICATION ATMOSPHERE | | | | | | | |
| INVESTIGATE CENTRIFUGAL CASTING TECHNIQUE | | | | | | | |
| MINIMIZE SPALLING IN CERAMIC LINERS | | | | | | | |

OBJECTIVES

Determine the physical and chemical stability of refractories in coal gasifier linings adjacent to the cold shell (i. e., in the 300-1000° F temperature range, at 100-1000 psi pressure, and in the presence of coal gasification atmospheres)

PROJECT JUSTIFICATION

The refractory lining of a gasification process vessel is a vital component needed to assure the reliable operation of the gasification process. Its function is mainly to prevent excessive heat losses, to keep the pressure vessel shell cool and to prevent corrosion of the shell. Degradation of the lining, to the point at which the hot gases come in contact with the shell, will cause a hot spot on the shell and an automatic shutdown of the unit. Since it is projected that second generation gasification plants will only have 2-4 gasifiers, this means that plant output will be reduced by 25-50 percent in the event of a shutdown. Thus, refractory lining failures must be prevented at all cost.

The Division of Fossil Energy Research materials program will cover most potential thermo-mechanical and hot corrosion problems expected by most refractory experts and coal conversion process design engineers. However, potential refractory problems in the lining adjacent to the metal shell of the process vessel have not been adequately covered. Degradation of the refractory lining in this area can potentially also be dangerous. The first lining of the CO₂ acceptor plant, Rapid City, S. D., failed completely after only a few days service due to a design failure. The vessel was relined with a more conventional lining material which has performed satisfactorily. This failure occurrence confirmed experience from the petrochemical industry that the lightweight insulating refractory must have a high enough crushing strength to prevent the outward movement of the hard facing material. It is conceivable that chemical degradation processes at the cold shell will reduce the strength of the insulating concrete to a point at which it no longer stops the hard facing material from moving outward. It is necessary to determine if this failure possibility is real and, if so, to identify materials which can be used to prevent this type of failure.

Chemical and Physical Stability of
Refractories for Use in Gasifiers

CONTRACT DATA

CONTRACT NO. E(11-1)-2904
CONTRACTOR University of Missouri
ADDRESS Rolla, Missouri 65401

PRIN. INVEST. D. E. Day
BEGIN/END 1 May 1976 thru 30 Apr 1978
CONTRACT VALUE \$121,385
SPONSOR FER
DIRECTORATE University Programs
TECH. PROJ. OFF. W. T. Bakker
CATEGORY/DETAIL Materials and Components/Materials
FUNDING
(thousands of dollars)

| FY | ERDA | CONTRACTOR | TOTAL |
|----|------|------------|-------|
| 76 | 121 | -- | 121 |

PROJECT DESCRIPTION

This investigation of refractories consists of the following four tasks:

- I. Prepare and characterize samples of various refractories for exposure to coal gasification environments, including: dense calcium aluminate bonded castables, dense phosphate bonded ram mixes, and lightweight castables. In addition to these materials, test at least one commercial product as a commercial reference.
- II. Expose the samples to various coal gasification atmospheres at 300-1000° F and 100-1000 psi pressure for at least 100 hr. Coal gasification atmospheres with various CO/CO₂ and H₂/H₂O ratios should be used, including the standard composition used in other ERDA programs. At high pressures and low temperatures, part of the H₂O will be in the liquid state. The samples must be exposed to both liquid and gaseous phases at these temperatures.
- III. Characterize the exposed samples by microstructural and chemical analysis and measurement of physical properties such as density, porosity, and cold and hot crushing strength.
- IV. Interpret and analyze all data. Describe failure mechanisms and make recommendations on the most desirable types of refractories for use in this application.

ACTIVITY SCHEDULE

| ACTIVITY | FY 76 | TRANS | FY 77 | FY 78 | FY 79 | FY 80 | FY 81 |
|----------------------------------|-------|-------|-------|-------|-------|-------|-------|
| PREPARE AND CHARACTERIZE SAMPLES | | | | | | | |
| TEST SAMPLES | | | | | | | |
| CHARACTERIZE EXPOSED SAMPLES | | | | | | | |
| ANALYZE DATA | | | | | | | |

OBJECTIVES

Develop Fe-base alloys having less than 10 percent of Cr and Ni with a corrosion rate less than 20 mils/yr in coal gasification atmosphere and with mechanical properties, fabricability, and weldability equal to 300 series austenitic stainless steels

PROJECT JUSTIFICATION

Work performed by the Metals Properties Council (MPC) has indicated that only very few high temperature alloys resist corrosion by coal gasification atmospheres containing 0.5 to 1.0 percent H₂S. Only high chrome alloys such as 316 stainless steel and 50 Cr-50 Ni alloy show little or no corrosion at 1800° F in a 1000 hr laboratory test, the 316 stainless steel only after being annealed. However, even these alloys corrode over longer periods of time at temperatures of 1500 to 1800° F in H₂S environments. Thus, presently available high temperature alloys, which were mainly developed because of their excellent oxidation resistance, may not be adequate structural materials in H₂S-rich coal gasification environments.

The MPC work generally indicates that the corrosion rate decreases with increasing chromium content. Thus, if a conventional alloy with an improved corrosion resistance can be developed, it will probably contain large amounts of chromium. This is undesirable from a commercial and political point of view. If the coal conversion industry develops as planned, a critical dependence on imported chrome ore could develop. The development of a low chrome alloy for use in coal conversion processes is a lengthy task and should be initiated as soon as possible.

Work done during the Korean War, when nickel was in short supply, indicated that aluminum and manganese additions to iron base alloys can be used as substitutes for chrome and nickel, respectively. Development of these alloys was dropped when nickel became more available after the Korean War, primarily because they lacked hot strength or were extremely difficult to weld and fabricate.

In the last 20 years our understanding of iron base alloys has greatly increased. It is believed that some of the techniques developed at the University of California to increase the hot strength of low alloy Cr-Mn steels can also be successfully applied to Fe-Mn-Al alloys. Strengthening of the alloy with intermetallic "laves" phases, followed by a patented spheroidization treatment, shows special promise. Available data indicate that very good alloys may be developed in either or both of two different ternary systems. The first is a ferritic class of alloys in the Fe-Cr-Al system that is low in Cr and high in Al. The second is an austenitic class of alloys in the Fe-Mn-Al system that is rich in Mn and high in Al. The key in both systems will be to balance structure and composition to achieve maximum stability and resistance to attack at temperatures up to 1800° F while maintaining useful mechanical behavior and properties. Control of additions of stabilizing elements most likely will be needed to achieve these results. An exploratory program to assure that coal gasification plants can be built with readily available materials appears fully justified at this time.

CONTRACT DATA

CONTRACT NO. E(49-18)-2299
 CONTRACTOR Lockheed Missiles and Space Co.
 ADDRESS 3251 Hanover Street
 Palo Alto, Calif. 94304
 PRIN. INVEST. R. A. Perkins
 BEGIN/END 14 May 1976 thru 23 Jan 1978
 CONTRACT VALUE \$298,431
 SPONSOR FER
 DIRECTORATE Materials and Power Generation
 TECH. PROJ. OFF. W. T. Bakker
 CATEGORY/DETAIL Materials and Components/Materials

FUNDING (thousands of dollars)

| FY | ERDA | CONTRACTOR | TOTAL |
|----|------|------------|-------|
| 76 | 298 | -- | 298 |

PROJECT DESCRIPTION

This materials development program consists of two primary tasks, each of which consists of a screening (S), a design and development (D), and an evaluation (E) phase.

- I. Design and develop an iron base alloy with improved sulfidation resistance. (S) Screen the Fe-Al-Mn systems (within the limits 5 to 20 percent Al, 0 to 40 percent Mn, balance Fe) for resistance to sulfidation using the standard coal gasification atmosphere used in other ERDA-sponsored programs. (D) Modify Fe-Al-Mn alloys having less than 20 mils/yr corrosion loss. Determine the effect of various combinations of Mn, Al, and Ta on sulfidation resistance. Improve the sulfidation resistance of Fe-Al-Mn alloys having 20 to 100 mils/yr corrosion loss by additions of Si and/or Cr (Si up to 5 percent, Cr up to 15 percent) as a back up study to the main effort of development of a chrome free alloy with a satisfactory combination of mechanical properties and corrosion resistance is doubtful. (E) Evaluate two or more of the most promising compositions over a wider range of environmental conditions to characterize more fully their potential as a replacement for currently used stainless steels and super alloys. Include corrosion-erosion testing in this evaluation.
- II. Design and develop an iron base alloy with properties at least equal to series 300 stainless steels, containing only minor quantities of Cr and Ni. (S) Map out the approximate structure-composition relationships for alloys in the Fe-Al-Mn system with 5 to 20 percent Al and 0 to 40 percent Mn. Measure hardness at room temperature and 1600° F. Correlate strength levels as inferred from hardness data with structure and composition. Select compositions for the Phase II design and development study in the range where these limits overlap those defined in Task I. (D) Further strengthen essentially Cr- and Si-free Al-Mn-Fe alloys by solid solution additions of Mo and additions of Mo or Ta, beyond their solubility limits in alpha iron to form stable Laves phases. Evaluate the alloys by tensile strength tests at room temperature and 1600° F, creep-rupture tests at 1600° F and room temperature "Charpy" impact tests. If preliminary results indicate that high Al sulfidation resistant alloys with acceptable physical properties cannot be developed, modify the system further by Si and Cr additions and evaluate in the same manner. (E) Characterize the physical properties of two or more of the most promising alloys more fully using long-term (1000 hr) creep tests at 1200 and 1800° F, tensile tests at 1200, 1600, and 1800° F, and determine room temperature, tensile, and brittle behavior after long-term (1000 hr) exposure to coal gasification atmospheres. Make a cursory evaluation of the weldability, fabricability, and producibility.

ACTIVITY SCHEDULE

| ACTIVITY | FY 76 | TRANS | FY 77 | FY 78 | FY 79 | FY 80 | FY 81 |
|-------------------------------------|-------|-------|-------|-------|-------|-------|-------|
| DESIGN AND DEVELOP IRON BASE ALLOYS | | | | | | | |
| IMPROVED SULFIDATION RESISTANCE | | | | | | | |
| EQUIVALENT TO SERIES 300 SS | | | | | | | |

Sulfidation Resistant Alloy for Coal Gasification Service

03-21-

Coal/Materials and Components/Materials Sulfur Resistant Alloy

Develop prototype equipment, techniques, and standards for in situ examination of thin coatings for bond quality, thickness, homogeneity, integrity, and any other condition that could lead to premature or unexpected failure.

PROJECT JUSTIFICATION

Thin protective coatings are frequently necessary to protect components, such as valves, ancillary parts of pressure vessels, and cyclones used in coal liquefaction and gasification systems. These coatings act as a barrier to protect the members from degradation brought about by erosion, corrosion, hydrogen embrittlement, etc., at elevated temperatures and pressures.

To ensure that a desired coating will maintain its protective properties throughout the life of the component, it is necessary to develop and apply nondestructive test (NDT) techniques for in situ examination. In addition, it is important to know when the coating has reached a point where it is no longer servicable so that it can be replaced before severe damage occurs. A variety of NDT techniques are required to accomplish these examinations because of the different types of coatings and substrates used.

Existing NDT equipment can be used in those areas where current technology has been developed. Where existing NDT equipment will not suffice, modifications to the equipment and techniques will be required or new equipment must be developed. In either case, the emphasis will be on continual monitoring during operations (i.e., a readout of the coating condition and change in condition as the process continues) so that process-induced coating failure can be detected before a major or critical component failure. This project will result in NDT equipment and procedures suited to ceramic, cermet, and metallic coatings bonded to components used in coal conversion processes.

CONTRACT NO. ORNL Project 7146
CONTRACTOR Oak Ridge National Laboratory
ADDRESS P.O. Box X
Oak Ridge, Tenn. 37830
PRIN. INVEST. R. W. McClung
BEGIN/END 1 Jan 1976 thru 31 Dec 1976
CONTRACT VALUE \$500,000
SPONSOR FER
DIRECTORATE Materials and Power Generation
TECH. PROJ. OFF. W. T. Bakker
CATEGORY/DETAIL Materials and Components/Materials

FUNDING (thousands of dollars)

| FY | ERDA | CONTRACTOR | TOTAL |
|----|------|------------|-------|
| 76 | 100 | -- | 100 |
| TQ | 100 | -- | 100 |

This two-phase study consists of the following tasks:

Phase I

- I. Obtain coated specimens of suitable size and shape for NDT determinations. The coating thicknesses are to range from 260 to 2600 μm in four approximately equal increments and should include $\text{B}_4\text{C} + \text{Co}$, $\text{TiC} + \text{Co}$, $\text{Cr}_3\text{C}_2 + \text{NiCr}$, $\text{Cr}_3\text{C}_2 + \text{NiAl}$, ZrO_2 , $\text{MgO} \cdot \text{ZrO}_2$, and $\text{Cr}_3\text{C}_3 + \text{NiCr}$ applied by plasma spraying on IN 800 or 316 stainless steel. As this work continues, investigate additional candidate coatings and bonding techniques.
- II. Conduct feasibility studies on the samples, with inspection methods that seem promising for ultimate use, i.e., in situ evaluation of coating in the aforementioned coal conversion processes. Demonstrate the relative capabilities and limitations for the different methods and their applicability to the coating materials. Select the best approach for development of inspection equipment and techniques.

This work comprises Phase I. The ERDA has the option of beginning Phase II, or discontinuing the contract if further work appears unpromising.

Phase II

Develop new prototype inspection techniques and equipment, ranging from application engineering with existing techniques to ensure adequate calibration and standardization of procedures to the development of new techniques and equipment. Apply the above work for in situ NDT of thin coatings. Specific inspection techniques to be resolved include: bond quality, coating thickness, homogeneity, and physical discontinuities.

ACTIVITY SCHEDULE

| ACTIVITY | FY 76 | TRANS | FY 77 | FY 78 | FY 79 | FY 80 | FY 81 |
|---|-------|-------|-------|-------|-------|-------|-------|
| OBTAIN COATED SPECIMENS | ----- | | | | | | |
| CONDUCT FEASIBILITY STUDIES OF TESTING TECHNIQUES | ----- | | | | | | |
| DEVELOP INSPECTION EQUIPMENT AND TECHNIQUES | | | ----- | | | | |
| APPLY TO IN SITU TESTING | | | | | | | |

Study conditions under which iron and nickel carbonyls are formed when CO is in contact with carbon and stainless steels

PROJECT JUSTIFICATION

Iron and nickel carbonyls are formed when CO gas contacts steels under certain conditions. These carbonyls are carried as a vapor unless they are decomposed at temperatures exceeding 200°C. In the PERC methanation work, if iron carbonyls are formed and carried as gas into the methanators containing nickel catalyst operating at 300° to 400° C, the carbonyls decompose, depositing iron on the surface of the nickel catalyst, causing the formation of carbon and changing the methanation catalyst to a Fischer-Tropsch catalyst.

There is no pertinent work in this area. This study is necessary for the development of the methanation step in coal gasification.

CONTRACT NO. ORNL Project 7154
CONTRACTOR Oak Ridge National Laboratory
ADDRESS P.O. Box X
Oak Ridge, Tenn. 37830

PRIN. INVEST. J. DeVan
BEGIN/END 1 Jan 1976 thru 31 Dec 1976
CONTRACT VALUE \$210,000
SPONSOR FER
DIRECTORATE Materials and Power Generation
TECH. PROJ. OFF. T. Cox (FER)/W. Michaels (PERC)
CATEGORY/DETAIL Materials and Components/Materials

FUNDING (thousands of dollars)

| FY | ERDA | CONTRACTOR | TOTAL |
|----|------|------------|-------|
| 76 | 110 | -- | 110 |
| TQ | 100 | -- | 100 |

Oak Ridge National Laboratory will investigate the formation of carbonyls under the following conditions:

Pressure: 20 to 70 atm total pressure

Atmosphere: Pure CO (no carbonyls) and synthetic gas (30 percent CH₄, 51 percent H₂, 17 percent CO, 2 percent CO₂, < 0.1 percent H₂O)

Temperature: 100 to 500° F

Vary contact residence time with the steels from 24 to 168 hr. Steels which exhibit no carbonyl formation under the longer time conditions may be subjected to extended contact time. Start tests with a commercial carbon steel and then proceed through the range of stainless steels and chromium steels. Select a method of determining the quantity of carbonyls produced.

ACTIVITY SCHEDULE

| ACTIVITY | FY 76 | TRANS | FY 77 | FY 78 | FY 79 | FY 80 | FY 81 |
|---|-------|-------|-------|-------|-------|-------|-------|
| SEARCH LITERATURE | — | | | | | | |
| ASSEMBLE AND CHECKOUT EQUIPMENT | — | — | | | | | |
| SCREEN COMMON STEELS | | — | — | | | | |
| DEVELOP AND DEMONSTRATE PREVENTION METHOD | | | — | — | | | |

Modify existing high temperature metal alloys for use in coal gasification atmospheres so that protective sulfide and carbide layers are formed instead of or in addition to the usual protective oxide scale.

PROJECT JUSTIFICATION

Studies by the Metals Properties Council (MPC) have indicated that very few high temperature alloys resist corrosion by coal gasification atmospheres containing 0.5 to 1.0 percent H_2S . Only high chrome alloys such as 310 stainless steel and 50 Cr-50 Ni alloy show little or no corrosion at 1800°F in a 1000 hr laboratory test, the 310 stainless steel only after being aluminized. It was further found that these alloys showed locally severe corrosion after longer periods at temperatures of 1500 to 1650°F in coal gasification atmospheres containing a variety of H_2S concentrations up to 1 percent. It is apparent that presently available high temperature alloys, which were mainly developed because of their excellent oxidation resistance, may not be adequate structural materials in H_2S rich coal gasification environments.

This potential problem can be attacked by redesigning the coal gasification reactor vessels so that internal metal parts are not required, developing sulfidation resistant coatings for existing alloys, and developing new sulfidation resistant alloys. These approaches all have advantages and disadvantages.

Development of metal alloys, which form better protective scales than existing alloys in coal gasification service appears to be the most direct and least costly approach, especially if this can be achieved through relatively minor changes to existing high temperature alloys. The general idea of oxide scale formation has been a subject of considerable research. However, research on the formation of protective carbide and sulfide layers has not been carried out in a systematic manner in the high temperature regimes expected to exist in coal gasification reactors.

The probability of successfully completing this study on the formation of carbide and sulfide protective coatings appears quite good, especially when combined with a thermodynamic study of known alloying elements capable of forming refractory carbides and sulfides.

CONTRACT NO. SL Project 7165
 CONTRACTOR Sandia Laboratories
 ADDRESS Livermore, Calif. 94550
 PRIN. INVEST. A. J. West
 BEGIN/END 1 Jan 1976 thru 30 Sep 1976
 CONTRACT VALUE \$450,000
 SPONSOR FER
 DIRECTORATE Materials and Power Generation
 TECH. PROJ. OFF. W. T. Bakker
 CATEGORY/DETAIL Materials and Components/Materials

FUNDING (thousands of dollars)

| FY | ERDA | CONTRACTOR | TOTAL |
|----|------|------------|-------|
| 76 | 75 | -- | 75 |
| TQ | 46 | -- | 46 |

This materials research project consists of the following seven tasks:

- I. Derive thermodynamic models for the prediction of elemental additions to alloys currently used in coal gasification systems that will allow formation of continuous carbide and sulfide films in the multicomponent coal gasification atmosphere. Prepare a list of promising alloy additions and develop several promising alloys using mild steel 20 Ni-25 Cr alloy (310), 50 Ni-50 Cr alloy, and nickel as base materials. Devise a statistical experimental design approach to confirm the capability of various alloying additions to provide useful protective scales.
- II. Design, construct, or adapt equipment for high temperature ($\pm 1900^\circ F$) corrosion testing of metals in coal gasification atmospheres at pressures to 150 psi. The required facilities shall have adequate space for exposure and storage of self-loaded specimens.
- III. Produce laboratory size melts of alloys selected from the list prepared in Task I. Prepare test specimens from the selected alloy melts. Subject the samples to appropriate heat treatments to optimize their physical properties and microstructure, and carburize or sulfurize or otherwise treat them to enhance their corrosion resistance. Measure their resistance to corrosion by coal gasification atmospheres at 1400 to 1800°F for a sufficient time to produce visible corrosion on at least one of the standard alloys.
- IV. Conduct microstructural investigations of the standard and experimental alloys before and after exposure to establish the rate of formation of protective layers as a function of temperature and gas composition, adherence and continuity of protective layers, and ability of protective layers to prevent sulfur and carbon embrittlement of the underlying metal. Characterize the protective layers to define extent of carbide and sulfide layers.
- V. Select the most promising alloys from Tasks III and IV for further long-term testing and evaluation.
- VI. Conduct tests similar to those described in Task IV. Perform standard ASTM tests to evaluate the mechanical properties of the newly developed alloys before and after exposure to coal gasification atmospheres.
- VII. Study and correlate thermodynamical models and experimental results to define a thermodynamical model that will predict the formation of protective scales of various alloys in a wide range of coal gasification atmospheres.

ACTIVITY SCHEDULE

| ACTIVITY | FY 76 | TRANS | FY 77 | FY 78 | FY 79 | FY 80 | FY 81 |
|--------------------------|-------|-------|-------|-------|-------|-------|-------|
| PREPARE PROMISING ALLOYS | | | | | | | |
| CONSTRUCT EQUIPMENT | | | | | | | |
| RUN CORROSION TESTS | | | | | | | |
| CONDUCT SUPPORTING TESTS | | | | | | | |
| PERFORM LONG-TERM TESTS | | | | | | | |
| CONDUCT SUPPORTING TESTS | | | | | | | |
| CORRELATE DATA | | | | | | | |

Determine the probability of alkali vaporization under the temperature, pressure, and atmospheric conditions occurring in various gasification processes

PROJECT JUSTIFICATION

It is well known that all coals contain considerable amounts of alkalis in various forms. The form in which the alkali occurs is less well known and also varies considerably for coals of different origin. Alkali corrosion and degradation of both refractories and metals is also well known. Alkali attack is the most common cause for refractory wear and failure in the blast furnace, especially in the bosh and lower stack areas, where the temperature ranges from 1700 to 2800°F. Thermodynamic data indicate that K_2O and Na_2O can be reduced by carbon or CO at temperatures above 1500°F. The reduction temperature is much higher for alkali silicates.

Since alkali metals vaporize at 1300 to 1650°F, reduction of the oxides results in vaporization. This vapor will be swept into the cooler region of the blast furnace or penetrate the lining and reoxidize. The oxides formed will react with CO_2 to form carbonates, or with silicates from the refractory lining to form alkali silicates. The formation of alkali silicates causes a large volume expansion which may lead to the destruction of the refractory and also lowers the softening point of the refractory significantly. Experience with large, coal fired boilers for electric power generation also indicates the volatilization of alkalis can occur under certain circumstances. In coal fired boilers, alkali-iron sulfates deposit on superheater tubes and cause both corrosion and fouling, i.e., the buildup of ash deposits on the tubes, which reduces the efficiency of heat transfer.

In summary, in the two major industrial applications of coal, alkali vaporization is not only proven but is also known to shorten the service life of the construction materials used and to decrease process efficiency. It is, therefore, likely that alkali volatilization followed by corrosion will be a factor in coal gasification vessels, especially those designed for high temperatures.

This project will study the alkali balance in both high- and low-Btu gasifiers in the 1500 to 3000°F range on a laboratory scale. This should permit the determination of the probability of alkali corrosion in various coal gasification processes.

Determination of the Probability of Alkali Degradation and Corrosion of Refractories and Metals in Various Coal Gasification Processes

CONTRACT NO. E(49-18)-2241
CONTRACTOR University of Missouri
ADDRESS Rolla, Mo. 65401

PRIN. INVEST. G. Lewis
BEGIN/END 1 Sep 1976 thru 31 Aug 1978
CONTRACT VALUE \$158,423
SPONSOR FER
DIRECTORATE Materials and Power Generation
TECH. PROJ. OFF. W. T. Bakker
CATEGORY/DETAIL Materials and Components/Materials

FUNDING (thousands of dollars)

| FY | ERDA | CONTRACTOR | TOTAL |
|----|------|------------|-------|
| 76 | 100 | 26 | 126 |

PROJECT DESCRIPTION

This research project consists of the following four tasks:

- I. Calculate the thermodynamic stability of oxides, sulfates, nitrates, carbonates, sulfides, cyanides, and silicates of sodium and potassium under the temperature, pressure, and atmospheric conditions existing in high and low Btu gasifiers.
- II. Conduct bench scale experiments to verify the thermodynamic calculations and to determine the rate of vaporization of selected alkali compounds such as potassium silicate, sodium sulfate, potassium cyanide, potassium chloride, and sodium nitrate, including a few high alkali coal ashes. Conduct all experiments using one typical high Btu and one typical low Btu gasification atmosphere in the presence of excess carbon in the form of char or coke at various pressures and temperatures under dynamic conditions.
- III. Conduct bench scale coal gasification experiments at 1800°, 2200°, and 2800°F to determine the rate of vaporization of alkali under dynamic conditions, using specified coals with a metallurgical grade blast furnace coke serving as the standard. Use oxygen-steam and air-steam mixtures to simulate high Btu and low Btu gasification atmospheres.
- IV. Correlate experimental and thermodynamic data and predict the degree of alkali vaporization for coals containing various amounts and forms of alkalis, when gasified at different temperature and pressures.

ACTIVITY SCHEDULE

| ACTIVITY | FY 76 | TRANS | FY 77 | FY 78 | FY 79 | FY 80 | FY 81 |
|---|-------|-------|-------|-------|-------|-------|-------|
| CALCULATE THERMODYNAMIC STABILITY | | | | | | | |
| VERIFY THERMODYNAMIC CALCULATIONS | | | | | | | |
| DETERMINE ALKALI VAPORIZATION | | | | | | | |
| CORRELATE EXPERIMENTAL AND THERMODYNAMIC DATA | | | | | | | |

Scale-up the process, in which SIALON is produced, using relatively inexpensive materials such as clay and coal

PROJECT JUSTIFICATION

The refractory lining of a slagging gasifier is probably the most critical component of a high Btu gasification plant since it is exposed to the combined action of erosion and corrosion by liquid slags at temperatures up to 3100°F. Most conventional oxide refractories are not stable in the presence of coal slags, but react to form low melting liquids and gradually wear away.

Experience with blast furnace bosh refractories, where erosion and corrosion by liquid slag is also the main wear mechanism, has shown that the refractory lining wears away relatively quickly, regardless of the refractory type used, until its hot face temperature is below the minimum temperature at which the refractory will react with the slag. At this point, further wear will cease, and the remaining refractory lining will be serviceable for many years, provided it is not too thin and no major mechanical upsets or cooling failures occur. This concept of the wear mechanism of refractory linings in blast furnaces has led to the use of carbon-graphite lining materials with a high thermal conductivity. It is believed that a similar refractory lining design concept is also valid for slagging gasifiers although the large quantities of steam in the gasifier atmosphere would oxidize carbon-graphite refractories.

Technically, a SIALON-based refractory would be ideal for this application. SIALON is a solid solution of silicon nitride and aluminum oxide, has a moderately high thermal conductivity, and an excellent slag resistance. Until recently SIALON was of little commercial value since it could only be made by hot pressing. Recently, Dr. Cutler has produced SIALON from clay and coal or char, using conventional ceramic manufacturing techniques, offering the possibility of an inexpensive class of refractories ideally suited for use in slagging gasifiers and many other applications where corrosion by acid slags is the main wear mechanism.

It appears worthwhile to demonstrate the technical and commercial feasibility of the University of Utah process to a point that commercial refractory producers can take over the project.

CONTRACT NO. E(49-18)-2407
 CONTRACTOR University of Utah
 ADDRESS Salt Lake City, Utah 84112
 PRIN. INVEST. I. B. Carter
 BEGIN/END 1 Jun 1976 thru 31 May 1978
 CONTRACT VALUE \$119,777
 SPONSOR FER
 DIRECTORATE University Programs
 TECH. PROJ. OFF. W. T. Bakker
 CATEGORY/DETAIL Materials and Components/Materials
 FUNDING
 (thousands of dollars)

| FY | ERDA | CONTRACTOR | TOTAL |
|----|------|------------|-------|
| 76 | 60 | -- -- | 60 |

This refractory development program consists of four tasks to:

- I. Investigate and define the essential process parameters needed to scale-up the production of dense SIALON grain from clay and coal. Areas to be investigated include: purity of raw materials, mineralogical composition of raw materials, fineness of raw materials, compaction of the raw materials mixture, and sintering of the green body.
- II. Produce a sufficient quantity of dense SIALON grain to prepare at least twenty-four 7 by 4.5 by 3 in. bricks.
- III. Prepare the 24 refractory bricks from the SIALON grain produced in Task II using standard refractory brick making procedures. Determine the optimum grain sizing, tempering, pressing, firing, and other process conditions. Optimize brick properties to provide the best combination of density, porosity, strength, and thermal shock resistance.
- IV. Characterize the SIALON brick produced, measuring the following properties: density, porosity, hot MOR (0 to 2900°F), thermal conductivity (0 to 2300°F), thermal and permanent linear changes (0 to 3100°F), and oxidation and abrasion resistance.

ACTIVITY SCHEDULE

| ACTIVITY | FY 76 | TRANS | FY 77 | FY 78 | FY 79 | FY 80 | FY 81 |
|----------------------------|-------|-------|-------|-------|-------|-------|-------|
| DEFINE PROCESS PARAMETERS | | | | | | | |
| PREPARE DENSE SIALON GRAIN | | | | | | | |
| PREPARE SIALON BRICKS | | | | | | | |
| CHARACTERIZE SIALON BRICKS | | | | | | | |

- Perform an experimental and theoretical study of the erosion of potential turbine materials by coal and ash particles to determine which factors are significant in such erosion
- Develop a computer model to facilitate the prediction of potential erosion in future turbomachinery design

PROJECT JUSTIFICATION

The conceptualization and development of high performance turbomachinery using pulverized coal would combine the mechanical simplicity of turbines with the use of an abundant fossil energy source. However, such turbine development has been forestalled by the obvious problems associated with the operation of equipment in a high velocity particulate stream.

The research to be performed in this study is experimental and theoretical and is designed to identify and order the parameters that affect the basic phenomena of erosion. The problems associated with the prediction of erosion in rotating machinery will be addressed by deriving and testing a theory of particle trajectory in a flow field with/after multiple impacts. Successful development of such a model could allow the development of turbines designed to minimize erosion by fluid mechanical considerations.

CONTRACT NO. E(49-18)-2465
 CONTRACTOR University of Cincinnati
 ADDRESS Cincinnati, Ohio 45221

PRIN. INVEST. W. Tabakoff
 BEGIN/END ~1 Jul 1976 thru 30 Jun 1979
 CONTRACT VALUE ~\$164,000
 SPONSOR FER
 DIRECTORATE University Programs
 TECH. PROJ. OFF. M. J. Biallas
 CATEGORY/DETAIL Materials and Components/Materials

FUNDING
(thousands of dollars)

| FY | ERDA | CONTRACTOR | TOTAL |
|----|------|------------|-------|
| | | | |

PROPOSAL NO. U60430SA

This study of the erosion of turbomachinery consists of four experimental and theoretical tasks to:

- I. Assemble and evaluate the previous research performed in the subject area.
- II. Use the University of Cincinnati wind tunnel to conduct an experimental program to determine the factors that are significant in influencing erosion by coal and ash under conditions that are representative of turbomachinery. Determine erosion characteristics on substrates chosen in consultation with ERDA, including, but not limited to, stainless steels, titanium, bonded composites, and other high temperature materials. Investigate the following variables: angle of impingement, particle velocity, particle size, surface properties, surface shape, particle hardness, and particle concentration.
- III. Use data obtained in Task II as a guide for the fabrication of a series of turbine guide vanes for which erosion characteristics shall be determined. Investigate the following variables: blade pitch, camber, and twist; solidity ratio; and inlet guide vane. In addition, investigate a fluid-mechanical approach for alleviating erosion in turbomachinery.
- IV. With the Task II and III data develop three models of particle migration and behavior in a turbine as they contribute to erosion. These models are: an erosion rate model, an erosion particle rebound model, and a quasi-analytical particle dynamic model. Develop a computer program using these models that will allow the prediction of the erosion in alternative turbomachinery designs.

ACTIVITY SCHEDULE

| ACTIVITY | FY 76 | TRANS | FY 77 | FY 78 | FY 79 | FY 80 | FY 81 |
|--|-------|-------|-------|-------|-------|-------|-------|
| SURVEY STATE OF THE ART | | | | | | | |
| DETERMINE FACTORS SIGNIFICANT IN INFLUENCING EROSION | | | | | | | |
| INVESTIGATE EROSION OF TURBINE BLADES | | | | | | | |
| DEVELOP PREDICTIVE MODELS AND COMPUTER PROGRAM | | | | | | | |

Collect, evaluate, and disseminate information pertinent to the use of materials and components in fossil energy systems

PROJECT JUSTIFICATION

Rapid development of fossil energy systems has placed demands on materials of construction, resulting in many research and development needs. The technology is new and complex, and this has created numerous problems with the use of materials and components in these systems.

It is important that good communications exist among those segments of the coal conversion and materials communities involved in the large-scale development of fossil energy systems. This is especially true of present and planned technical activities to evaluate the performance, analyze failures, and develop improved materials and components for these systems. While results of current technical activities can be found in scientific and technical reports, rapid dissemination of the most pertinent information can be accomplished most effectively through a periodic newsletter. Such exchanges of information or experiences can reduce or eliminate duplicate efforts and result in a savings of both developmental time and money.

In 1975, ERDA contracted with Battelle to prepare a quarterly "Newsletter on Materials and Components in Fossil-Energy Applications." To date, four newsletters have been prepared and distributed to government, industry, and academic personnel involved in fossil-energy development.

The newsletters issued so far have been very well received by both the materials and the coal conversion communities. The mailing list includes about 1100 recipients in industry, universities, and various government agencies concerned with energy or materials. Continuation of the newsletter appears justified since it is the main vehicle for disseminating materials information pertinent to coal conversion and utilization technology.

CONTRACT NO. E(49-18)-1804 (Mod)
 CONTRACTOR Battelle Memorial Institute
 ADDRESS 505 King Avenue
 Columbus, Ohio 43201
 PRIN. INVEST. J. R. Schorr
 BEGIN/END 21 Mar 1975 thru 20 Mar 1979
 CONTRACT VALUE \$355,200
 SPONSOR FER
 DIRECTORATE Materials and Power Generation
 TECH. PROJ. OFF. H. E. Frankel
 CATEGORY/DETAIL Materials and Components/Materials

FUNDING (thousands of dollars)

| FY | ERDA | CONTRACTOR | TOTAL |
|----|------|------------|-------|
| 75 | 54 | -- | 54 |
| 76 | 91 | -- | 91 |

This project to disseminate materials and components information related to fossil energy systems consists of five tasks to:

- I. Deliver a draft copy for review and approval by ERDA not later than 7 weeks after contract start. The scope and format will be determined jointly with ERDA, but is expected to follow the pattern already established.
- II. Deliver a camera-ready copy to the Science Service Branch of the Office of Public Affairs of ERDA or person(s) designated by this office within 14 days after ERDA's approval.
- III. Collect, screen, and store the information pertinent to the preparation of the newsletter.
- IV. Maintain and update a computer-based mailing list. Supply this list to ERDA along with the camera-ready copy.
- V. Repeat Tasks I and II at 2-month intervals, resulting in the preparation of 18 issues of the newsletter during the 3-year contract period.

ACTIVITY SCHEDULE

| ACTIVITY | FY 76 | TRANS | FY 77 | FY 78 | FY 79 | FY 80 | FY 81 |
|---------------------|-------------|-------|-----------------------------|-------|-------|-------|-------|
| PUBLISH NEWSLETTERS | △ △ △ △ △ △ | | △ △ △ △ △ △ △ △ △ △ △ △ △ △ | | | | |

Discuss current ceramic material problems in energy conversion systems and recommend future research in this area

PROJECT JUSTIFICATION

The coal gasification environments and the size of the process equipment expected are sufficiently different from existing processes and process equipment to justify research and development on ceramic materials for coal conversion processes. These materials will be needed both for thermal insulation and to protect structural metal parts from erosion and corrosion.

Successful implementation of a ceramics R&D program will require inputs from qualified ceramists from industry and academia on materials problems and requirements in coal conversion processes. This Workshop will provide the inputs and will also serve to familiarize the ceramic materials community with the energy conversion field, thus enlarging the field of knowledgeable contractors ERDA can draw on when future needs arise.

CONTRACT NO. E(49-18)-2209
 CONTRACTOR Battelle Memorial Institute
 ADDRESS 505 King Avenue
 Columbus, Ohio 43201
 PRIN. INVEST. J. R. Schorr
 BEGIN/END 8 Oct 1975 thru 27 Apr 1976
 CONTRACT VALUE \$7645
 SPONSOR FER
 DIRECTORATE Materials and Power Generation
 TECH. PROJ. OFF. W. T. Bakker
 CATEGORY/DETAIL Materials and Components/Materials

FUNDING (thousands of dollars)

| FY | ERDA | CONTRACTOR | TOTAL |
|----|------|------------|-------|
| 76 | 8 | -- | 8 |

PROJECT DESCRIPTION

This project to inform the ceramics community regarding coal conversion material problems consists of three tasks to:

- I. Organize a Workshop in which ceramic scientists and engineers from industry and academia are brought together with the developers of new energy conversion systems to discuss requirements for ceramic materials in those systems.
- II. Prepare a Workshop report consisting of the proceedings of each session, including conclusions and research recommendations.
- III. Distribute 600 copies of the Workshop report to the participants and to ERDA/FE for distribution to interested parties.

ACTIVITY SCHEDULE

| ACTIVITY | FY 76 | TRANS | FY 77 | FY 78 | FY 79 | FY 80 | FY 81 |
|-------------------------------|-------|-------|-------|-------|-------|-------|-------|
| ORGANIZE WORKSHOP | | | | | | | |
| PREPARE REPORT OF PROCEEDINGS | | | | | | | |
| DISTRIBUTE REPORT | | | | | | | |

Critically survey and evaluate existing literature and information from industry sources on high temperature oxidation and corrosion of metals and alloys in electrical generating systems and publish the results in handbook form

PROJECT JUSTIFICATION

High temperature metal corrosion will be a significant factor affecting the reliability and cost of most of the coal conversion and direct utilization projects presently considered by ERDA. To guide the Division of Fossil Energy Research materials research efforts; it is necessary to be thoroughly familiar with high temperature corrosion problems in related industries, such as electric power generation, especially that segment using coal-fired boiler systems. A critical review of the literature in this area is needed. Since ERPI also feels the need for such a survey and review, it will share half the contract cost and provide the technical manpower to supervise the project.

CONTRACT NO. E(49-18)-2266
CONTRACTOR Electric Power Research Institute
ADDRESS P.O. Box 10412
Palo Alto, Calif. 94304

PRIN. INVEST. R. I. Jaffee
BEGIN/END 8 Apr 1976 thru 7 Jul 1977
CONTRACT VALUE \$18,000
SPONSOR FER
DIRECTORATE Materials and Power Generation
TECH. PROJ. OFF. W. T. Bakker
CATEGORY/DETAIL Materials and Components/Materials

FUNDING (thousands of dollars)

| FY | ERDA | CONTRACTOR | TOTAL |
|----|------|------------|-------|
| 76 | 18 | -- | 18 |

This literature review project consists of the following tasks:

- I. Search the government literature using the MCIC files and computer access facilities at Battelle. British and European government literature will be covered from Liverpool.
- II. Search the technical and scientific literature, both at Battelle and Liverpool. Search the Russian and Eastern European literature at Battelle. Read papers and investigate references.
- III. Request information from applicable industrial organizations or access to their internal reports. Where necessary, follow up this initial request by a visit to particular organizations to elicit information.
- IV. Organize the available data in the form of a summary subdivided according to the specific alloy, with cross-reference to other relevant data. Send this to a number of experts for their comments.
- V. Prepare the Handbook.

ACTIVITY SCHEDULE

| ACTIVITY | FY 76 | TRANS | FY 77 | FY 78 | FY 79 | FY 80 | FY 81 |
|-------------------------------|-------|-------|-------|-------|-------|-------|-------|
| SEARCH GOVERNMENT LITERATURE | | | | | | | |
| SEARCH TECHNICAL LITERATURE | | | | | | | |
| OBTAIN INDUSTRIAL INFORMATION | | | | | | | |
| ORGANIZE DATA | | | | | | | |
| PREPARE HANDBOOK | | | | | | | |

Preparation of a Data Book on High Temperature Oxidation and Corrosion of Metals and Alloys in Electrical Generation Systems

03-30

Coal/Materials and Components/Materials
High Temperature Corrosion

Project Title: Alternate Materials of Construction for Geothermal Applications

Contract No: E(30-1)-0016

Contractor: Brookhaven National Laboratory
Upton, New York

Principal Investigators: M. Steinberg
(516) 345-2123, Ext. 3036
FTS 664-3036

L. E. Kukacka
(516) 345-2123, Ext. 3065
FTS 664-3065

Program Manager: Robert R. Reeber
(202) 376-4910
FTS 376-4910

Project Objective: To test several energy-related applications of concrete polymer materials as potential solution of corrosion problems.

Funding: FY 75 - \$ 99K
FY 76 - \$150K

Contract Term: 7/1/74-6/30/76

Reports Issued: M. Steinberg, "Concrete-Polymer Composite Materials Development." Proc. of Third Inter-Amer. Conf. on Materials Tech., Rio de Janeiro, Aug. 1972, H. H. Frye and C. J. McHargue, Editors, pp. 555-9.

Project Title: Alternate Materials of Construction for Geothermal Applications

Contract No: E(30-1)-0016

SUMMARY

The concrete-polymer composite materials developed at BNL and funded by the Office of Saline Water also appear to offer an economical approach to the problem of handling hot geothermal brines.

Laboratory formulation studies, long term durability tests, and limited field tests have been made over several years for materials to withstand brine at temperatures up to 175 degrees C (350 degrees F).

The program tasks are listed below:

Task 1 -- Selection of Lining Materials: A survey will be performed to develop polymer-aggregate systems that can withstand temperatures up to 300 degrees C. Initial work will be with highly crosslinked styrene and acrylate systems.

Task 2 -- Process Technology for PC Linings: Based upon the results obtained in Task 1, PC formulations will be developed and techniques for placing protective linings on steel pipe and plate tested.

Task 3 -- Physical and Chemical Property Testing: The suitability of materials and linings formulated in Tasks 1 and 2 will be evaluated for physical and chemical stability under geothermal use conditions.

Tests of materials formulated in Task 1 are underway at four geothermal sites. Preliminary results are very good with samples retaining high strength after 6 months exposure to geothermal brines and steam at temperatures up to 238 degrees C (460 degrees F). Under Task 2 many types of pipe lining have been fabricated and are under test. Work on Task 3 is progressing in the laboratory.

Project Title: Cementing of Geothermal Wells

Contract No: E(30-1)-0016

Contractor: Brookhaven National Laboratory
Upton, New York

Principal Investigators: M. Steinberg
(516) 345-2123, Ext. 3036
FTS 664-3036

L. E. Kukacka
(516) 345-2123, Ext. 3065
FTS 664-3065

Program Manager: Robert R. Reeber
(202) 376-4910
FTS 376-4910

Project Objective: Evaluate potential of various polymer cements for geothermal wells and coordinate a well cementing research program definition study.

Funding: FY 75 - \$
FY 76 - \$ 20K

Contract Term: 2/1/76-6/30/76

Reports Issued: None

Project Title: Cementing of Geothermal Wells

Contract No: E(30-1)-0016

SUMMARY

This project is being conducted to evaluate current status of geothermal well cementing capability and to coordinate a test program to develop adequate geothermal well cementing capability for insuring a maximum predicted well life.

A variety of polymer cement compositions developed at Brookhaven will be tested. Preliminary shallow well cementing indicate this type of material can be placed and cured under water.

Project Title: Corrosivity of Geothermal Brines

Contract No: W-7405-ENG-26

Contractor: Oak Ridge National Laboratory
Oak Ridge, Tennessee

Principal Investigator: John Griess
(615) 483-8611, Ext. 1546
FTS 850-1546

Program Manager: Robert R. Reeber
(202) 376-4910
FTS 376-4910

Project Objective: To develop the means to estimate the corrosivity of brines from a knowledge of their composition and temperature.

Funding: FY 75 -- \$ 54K
FY 76 - \$175K

Contract Term: 7/1/74-6/30/76

Reports Issued: None

Project Title: Corrosivity of Geothermal Brines

Contract No: W-7405-ENG-26

SUMMARY

The project will seek to gain an understanding of how the individual constituents affect corrosion and determine synergistic effects, if any. To accomplish this, a data base will be established, by electrochemical methods, on the influence of major and minor constituents of geothermal brines on the corrosion of candidate metallic materials of construction. The influence of scale growth on corrosion processes will be investigated. A quantitative evaluation will be made of the effect of solution flow on corrosion kinetics of promising metals at high temperatures and pressures. Field testing of the most promising materials will be carried out.

Laboratory equipment is being assembled for continuous operation and automated data recording. Preliminary runs using batch operation show good capability of controlling chemical and physical parameters in the test loop.

Project Title: Investigate Geothermal Corrosion--Study of Factors
Limiting Use of Iron Base Alloys vs Alternate
Materials in Mildly Acidic Geothermal Waters and
Steam

Contract No: E(45-1)-1830

Contractor: Battelle Pacific Northwest Laboratory
Richland, Washington

Principal Investigator: D. W. Shannon
(509) 942-3139
FTS 444-3139

Program Manager: Robert R. Reeber
(202) 376-4910
FTS 376-4910

Project Objective: To clarify corrosion factors which influence
materials selection for geothermal power plants.

Funding: FY 75 - \$ 60K
FY 76 - \$228K

Contract Term: 7/1/74-6/30/76

Reports Issued: D. W. Shannon, "Economic Impact of Corrosion and
Scaling Problems in Geothermal Energy Systems,"
Battelle Pacific Northwest Laboratory report
BNWL-1866 (January 1975)

Project Title: Investigate Geothermal Corrosion--Study of Factors Limiting Use of Iron Base Alloys vs Alternate Materials in Mildly Acidic Geothermal Waters and Steam

Contract No: E(45-1)-1830

SUMMARY

The major effort in FY 1976 will be directed to a systematic study of factors affecting carbon steels in mildly acidic solutions up to 250°C (480°F), completion of the first corrosion fatigue test, fabrication of the field corrosion test unit and preparing a suitable field site.

Initial efforts, which will establish corrosion limits to the use of iron base alloys in geothermal water, will utilize laboratory scale stirred autoclaves (1 to 6 liter) to investigate the high temperature, high pressure corrosion behavior of iron base alloys in weakly acidic fluids and compare performance with alternate materials such as aluminum, titanium, and nickel 16 Cr - 16 Mo alloys.

To investigate corrosion fatigue in geothermal steam, a stirred autoclave will be set up to produce cyclic stress in specimens of 12% Cr steel which is in general use as a thermal turbine blade material. By varying the partial pressures of H_2 , H_2S , and H_2O and traces of O_2 the chemical activity of the steam will be varied. Time to failure will be monitored by electrical connections to the specimens.

Finally, a field corrosion test will measure corrosion in geothermal water typical of waters proposed for binary cycle plants. Ideally a mildly acidic water with a pH around 5.5 to 6 would be chosen. After base line data are obtained on corrosion in the normal well water, changes in pH, temperature, and velocity will be made in the water flowing through the test stand. The change in corrosion performance will be compared with initial data to establish the validity of the conclusions obtained with the laboratory scale autoclaves.

Work began in April 1975. Detailed planning began and supplies for the R&D program were ordered. Existing ERDA autoclave equipment at PNL was relocated and several scouting tests of carbon steel in carbonic acid/bicarbonate solutions were run at temperatures up to 250°C. Work began on setting up and calibrating analytical chemistry procedures. Planning began on the experimental design for the corrosion fatigue test. Survey work began to locate a suitable site for the field test.

Work on this project has been delayed due to a labor dispute.

Project Title: Develop Standard Methods and Manual for Sampling
and Analysis for Geothermal Fluids and Gases

Contract No: E(45-1)-1830

Contractor: Battelle Pacific Northwest Laboratory
Richland, Washington

Principal
Investigator: D. W. Shannon
(509) 942-3139
FTS 444-3139

Program Manager: Robert R. Reeber
(202) 376-4910
FTS 376-4910

Project Objective: Develop standard methods and publish a manual for
sampling and analysis of geothermal fluids and
gases in order to assure accuracy, reliability,
and intercomparability of reported results.

Funding: FY 75 - --
FY 76 - \$ 50K

Contract Term: 7/1/75-6/30/76

Reports Issued: None

Project Title: Develop Standard Methods and Manual for Sampling
and Analysis for Geothermal Fluids and Gases

Contract No: E(45-1)-1830

SUMMARY

Development of standard methods and publication of a manual for sampling and analysis of geothermal fluids will assist the developing geothermal industry to meet its analytical needs by reducing the analytical methods research required by each organization.

Work in FY 1976 will consist of assembling the state-of-the-art laboratory and field methods and planning a systematic test and evaluation program. The existing methods for sampling and analysis now used by various investigators will be assembled and critiqued by an analytical standards group of three user institutions. In order to accelerate standardization as soon as possible this "collection" of methods will be published for comment and interim use on a voluntary basis. The selection of material for this first report will reflect the standard group's best judgment on candidate methods.

Work on this project was begun in February 1976 and is still in the formative stages.

Project Title: The Development and Evaluation of Elastomeric
Materials for Geothermal Applications

Contract No: E(49-27)-1011

Contractor: NASA
Pasadena, California

Work Performed by: Jet Propulsion Laboratory
California Institute of Technology
Pasadena, California

Principal Investigator: William A. Mueller
(213) 354-3073
FTS 792-3073

Program Manager: Robert R. Reeber
(202) 376-4910
FTS 376-4910

Project Objective: Modify elastomeric compounds to extend their
capabilities to include service in geothermal
environments.

Funding: FY 75 - --
FY 76 - \$104K

Contract Term: 6/21/76-6/30/77

Reports Issued: None

Project Title: The Development and Evaluation of Elastomeric
Materials for Geothermal Applications

Contract No: E(49-27)-1011

SUMMARY

In this project, techniques will be developed to modify elastomeric compounds to extend their capabilities to include service in geothermal environments. Specifically, one material will be developed for O-ring seals for 24-hour service at 260 degrees C in a geothermal environment. One goal will be the development in 1977 of at least one elastomer for use as an O-ring seal at 300 degrees C under high pressure in a geothermal environment.

A state-of-the-art survey of elastomeric materials for O-rings, seals, packing, etc., suitable for high temperature (260 degrees C) operation in geothermal environments, has been initiated.

Project Title: Materials Evaluation and Development for Sour
Environments

Contract No: E(11-1)-2602

Contractor: Case Western Reserve University
Cleveland, Ohio

Principal Investigator: A. R. Troiano
(216) 368-4234

Program Manager: Robert R. Reeber
(202) 376-4910
FTS 376-4910

Project Objective: To adapt existing materials to new uses in sour
environments.

Funding: FY 75 - \$325K
FY 76 - \$ 12K

Contract Term: 12/15/76-5/31/77

Reports Issued: None

Project Title: Materials Evaluation and Development for Sour
Environments

Contract No: E(11-1)-2602

SUMMARY

Samples of two classes of commercially available alloys are being subjected to a range of environments chosen to produce stress corrosion cracking. Those alloys found to be resistant will be processed by thermal and mechanical treatments to further improve their susceptibility to stress corrosion cracking.

Screening tests for both commercial constructional and high strength corrosion resistant alloys are in progress at both ambient and higher temperatures to 290 degrees C (550 degrees F).

Work on improving heats of commercially suitable alloys is being coordinated with Armco Steel Company.

Project Title: Scale Formation and Control

Contract No: W-7405-ENG-48

Contractor: Lawrence Livermore Laboratory
Livermore, California

Principal Investigator: G. E. Tardiff
(415) 447-1100, Ext. 3946
FTS 457-3946

Program Manager: Robert R. Reeber
(202) 376-4910
FTS 376-4910

Project Objective: To develop and evaluate problems of scale formation from geothermal deposits, particularly the hot brines such as those of the Salton Trough in California.

Funding: FY 75 - \$600K
FY 76 - \$900K

Contract Term: 7/1/74-6/30/76

Reports Issued: A. Austin, G. Higgins, J. Howard, "The Total Flow Concept for Recovery of Energy from Geothermal Hot Brine Deposit," Lawrence Livermore Laboratory report UCRL-51366 (April 1973)

D. D. Jackson, J. H. Hill, "Possibilities for Controlling Heavy Metal Sulfides in Scale from Geothermal Brines," Lawrence Livermore Laboratory report UCRL-51977 (January 1976)

R. N. Schock, A. Duba, "The Effect of Electrical Potential on Scale Formation in Saline Sea Brine," Lawrence Livermore Laboratory report UCRL-51944 (November 1975)

L. B. Owen, "Precipitation of Amorphous Silica from High-Temperature Hypersaline Geothermal Brines," Lawrence Livermore Laboratory report UCRL-51866, to be presented at the 1976 Spring Annual Meeting and Journal of Volcanology, Geochemistry, and Petrology.

Project Title: Scale Formation and Control

Contract No: W-7405-ENG-48

SUMMARY

The purpose of the project is study of scale formation control for brines, particularly the hot brines such as those of the Salton Trough in California.

Fluid handling and materials studies involve investigations of the flow and chemistry of the geothermal fluids in the reservoir through the production well, through the energy conversion apparatus and on to disposal. Understanding the behavior of structural materials in the brine environment is included in this category. Special problems of the hot brines of the Salton Trough are scaling and corrosion. The details of scale formation and corrosion will be investigated.

A field testing laboratory was built and moved to the Imperial Valley. The test facility is being used to collect data on chemical modifications of brine to control scaling.

Project Title: Development of Probes for Down Hole and In-Line
Chemical Analysis of High Pressure, High Temperature
Geothermal Fluids

Contract No: E(45-1)-1830

Contractor: Battelle Pacific Northwest Laboratory
Richland, Washington

Principal Investigator: D. W. Shannon
(509) 942-3139
FTS 444-3139

Program Manager: Robert R. Reeber
(202) 376-4910
FTS 376-4910

Project Objective: Develop probes to monitor high temperature, down
hole, geothermal fluid chemistry.

Funding: FY 75 - --
FY 76 - \$ 70K

Contract Term: 7/1/75-6/30/76

Reports Issued: None

Project Title: Development of Probes for Down Hole and In-Line
Chemical Analysis of High Pressure, High Temperature
Geothermal Fluids

Contract No: E(45-1)-1830

SUMMARY

This project will initiate high temperature electrochemical studies of geothermal fluids in support of probe development and probe data interpretation and will develop a basis for control of geothermal fluid chemistry to minimize deposition in plant and during reinjection into the reservoir.

Measurement of pH, oxidation potential, solution conductivity and corrosion in situ will provide a satisfactory basis of understanding of working fluid chemistry. Present methods of monitoring geothermal fluid chemistry after cooling and depressurization do not give an adequate picture of local chemistry in the reservoir or in the plant. Successful development of even simple probes for use down hole or in the plant will improve understanding of what is happening internally during operation and provide a basis for correction of the problem.

Work in FY 1976 will be focused establishing the feasibility of high temperature, high pressure chemical measurements. The bulk of the effort will be focused on development of suitable high temperature electrodes to measure E_h , pH, and conductivity in a high pressure geothermal environment.

This project started late in the fiscal year. Accomplishments include the design of candidate measurement systems and coordination with other workers in the field.

Project Title: Silica Precipitation and Brine Management

Contract No: W-7405-ENG-48

Contractor: Lawrence Berkeley Laboratory
Berkeley, California

Principal Investigators: John A. Apps
(415) 843-2740 ext. 5193
FTS 451-5193

Oleh Weres
(415) 843-2740 ext. 5625
FTS 451-5193

Program Manager: Robert R. Reeber
(202) 376-4910
FTS 376-4910

Project Objective: To characterize the chemical compositions of
geothermal brines and to develop a geothermal brine
management plan.

Funding: FY 75 - --
FY 76 - \$132K

Contract Term: 4/1/76-6/30/76

Reports Issued: None

Project Title: Silica Precipitation and Brine Management

Contract No: W-7405-ENG-48

SUMMARY

The objectives of this project are to characterize the chemical compositions of geothermal brines, to assist in the development of a geothermal brine management plan, and to develop a model for use in computer simulations of geothermal plant performance degradation.

The development of a brine management program plan has been initiated, as has acquisition and cataloging of available chemical data for brines likely to be exploited during the next 10 years.

Project Title: Precipitation and Scaling in Dynamic Geothermal Systems

Contract No: W-7405-ENG-26

Contractor: Oak Ridge National Laboratory
Oak Ridge, Tennessee

Principal Investigator: E. G. Bohlman
(615) 483-8611, Ext. 31371
FTS 850-1371

Program Manager: Robert R. Reeber
(202) 376-4910
FTS 376-4910

Project Objective: Experimentation with synthetic brines under various conditions.

Funding: FY 75 - \$ 85K
FY 76 - \$250K

Contract Term: 7/1/74-6/30/76

Reports Issued: None

Project Title: Precipitation and Scaling in Dynamic Geothermal Systems

Contract No: W-7405-ENG-26

SUMMARY

Conditions for precipitation and scaling in dynamic geothermal systems will be determined by modifying an existing 100 gpm titanium loop.

Solids formed under varying flow conditions, temperature (to 300 degrees C), and brine compositions will be quantitatively characterized by chemical analysis, metallography, crystallography and electron microscopy. This loop demonstrated excellent serviceability over a period of years in saline water corrosion studies with and without pollutant additives such as H_2S , NH_3 , and SO_2 and should be equally satisfactory in this application.

Preliminary experiments, varying temperature and pH in a precursor once-through system, have produced scale that consists of conglomerates of particles cemented to the system wall. The scale has been characterized and is similar to that formed in geothermal brines.

Project Title: Study of Silica Scaling from Geothermal Brines

Contract No: E(11-1)-2607

Contractor: EIC Incorporated
Newton, Massachusetts

Principal Investigator: W. W. Harvey
(617) 965-2710

Program Manager: Robert R. Reeber
(202) 376-4910
FTS 376-4910

Project Objective: To establish kinetics and mechanisms of initial stages of silica scale formation in synthetic brines and indicate ways to control scale formation in geothermal power plants.

Funding: FY 75 - \$289K
FY 76 - --

Contract Term: 11/15/74-4/30/77

Reports Issued: None

Project Title: Study of Silica Scaling from Geothermal Brines

Contract No: E(11-1)-2607

SUMMARY

Homogeneous nucleation and growth of silica from brines supersaturated in silicic acid have been studied over a range of pH(4.5-6.7), temperature (75-105 degrees C), salinity, and silicic acid concentrations (700 to 200 ppm as SiO_2).

The isothermal rate of SiO_2 condensation is a strong function of supersaturation ($c/c_{eq.}$), pH, and salinity. Overall kinetics follow the general theory of phase transitions and are adequately described by the Volmer expressions for condensation from solution. At supersaturations less than about 3 an induction period is observed; it amounts to several hundred minutes at supersaturations of 2. The "critical" supersaturation is estimated to be near 1.7.

Primary nuclei have radii of the order of a few Angstroms. Particles with radii of several hundred Angstroms are obtained at equilibrium. Nuclei growth is activation controlled, at least initially. Growth kinetics near equilibrium appear to be diffusion controlled.

The substantial effect of brine salinity on condensation rate can be quantitatively accounted for by decreased equilibrium solubility of H_4SiO_4 with increasing salt content.

An increase of one pH unit in the range from 4.5 to 6.7 decreases the induction period by a factor of about 10. The pH dependence of the growth rate is consistent with the hypothesis that H_4SiO_4 is one of the reacting species. Changes of temperature (75 to 105 degrees C) have little effect either on nucleation or growth rate at a fixed initial H_4SiO_4 concentration.

This result suggests that the rate increase at the higher temperatures is compensated by lower supersaturation at the higher temperatures.

Future work will include:

- (A) effects of temperature (105 to 225 degrees C) and of cations (Fe, Al, and other metal ions) on nucleation and growth
- (B) heterogeneous nucleation on various surfaces (stainless steel, Ti, non-metallics)
- (C) Correlation of scale composition to solution composition

A scale management conference was organized for August 2-4, 1976.

Project Title: A Study of Scale Formation and Suppression in Heat
Exchange Systems for Geothermal Brines

Contract No: E(11-1)-2833

Contractor: Dow Chemical, USA
Freeport, Texas

Principal Investigator: John S. Wilson
(713) 238-4153

Program Manager: Robert R. Reeber
(202) 376-4910
FTS 376-4910

Project Objective: To investigate techniques for controlling scale
formation from geothermal brine.

Funding: FY 75 - --
FY 76 - \$112K

Contract Term: 1/12/76-4/5/77

Reports Issued: None

Project Title: A Study of Scale Formation and Suppression in Heat
Exchange Systems for Geothermal Brines

Contract No: E(11-1)-2833

SUMMARY

Experiments will be conducted to produce controlled scaling in heat exchangers from simulated geothermal brine. Four techniques will be studied to control scale formation; maintain high pressure of carbon dioxide, add surface active polymers, remove calcium by the addition of sulfate and electromagnetic protection. Work has commenced on installation of small heat exchangers for electromagnetic protection experiments. The work is being done at the Dow Chemical water materials test center in Freeport, Texas, and uses existing equipment of the Office of Saline Water.

INPUT TO EMACC ANNUAL REPORT

Division of Conservation Research and Technology

The Division of Conservation Research and Technology had responsibility for materials R&D in support of energy conversion and storage systems in FY 1976. (In June 1976, a new Division of Energy Storage Systems was formed.) In the energy conversion category, the largest effort was on high temperature structural materials, including advanced ceramics, for gas turbines and heat exchangers. Individual contracts included work on processing and joining of ceramics, material property evaluation, and oxide thermal barrier coating development for super-alloys.

A second major effort in energy conversion was the development of electrocatalysts, electrolytes, and other materials for fuel cells. Materials for first generation acid electrolyte fuel cells was of foremost interest, but some attention was also given to high temperature solid electrolytes for advanced systems.

Assessment studies were initiated in the area of materials recycling, metals and plastics in particular. Serious thoughts were given to expanding the assessment study on plastics to a broader study on the increased use of polymeric materials in energy systems.

Since the Vice Chairman and project leader (J. H. Swisher) of the COMAT Energy Task Group was a member of the Division, the COMAT study on materials R&D needs in the entire energy program was a significant activity for all the materials staff. A report on the near-term program has been approved for publication.

In the energy storage category, most of the materials R&D was an integral part of engineering projects. The best example is the high temperature battery program, in which beta alumina development, and separator and containment material evaluation received a great deal of effort. In other storage projects, a significant effort was placed on hydrogen compatibility of alloys for storage vessels and pipelines, polymer-matrix composites for flywheels, and media for thermal energy storage.



J. H. Swisher

Overview

Materials represent one of the major long-lead time problem areas for power producing fusion systems. Fusion reactors present their own special materials problems related to, but often separate from, those of LMFBR's and other systems. The most severe of these deals with the effects of 14 MeV neutrons and other high energy particles generated in fusion reactors. This radiation not only results in increased bulk radiation effects (displacement damage, void formation, helium embrittlement) but gives rise to a whole new set of surface related effects including neutron and charged-particle sputtering, blistering, and chemical sputtering. Such effects are known to exist, but there is only a modest knowledge about their magnitude, reproducibility, variability with materials, and energy dependence. Other areas that require investigation include potential coolant and blanket materials, compatibility with structural components, welding and fabrication techniques, nondestructive testing, high temperature design, and adequate electrical insulators. A part of the DMFE materials program is the development of radiation facility concepts for testing of materials and components.

The objectives of the program are to develop the materials and materials technology for commercial fusion power generation. This includes the development of new radiation resistant first wall/structural alloys, as well as the development and testing of other materials such as insulators, advanced superconductors, moderator and breeding materials, and materials for power-conversion systems. The general requirements have been assessed in various system studies as well as by the DMFE Materials and Radiation Effects Branch and are being incorporated into a program plan. Task groups are being formed to assess the detailed technical requirements for commercial reactors and intermediate systems and to establish tasks and milestones to meet them.

The DMFE Reactor materials program is divided into five research and development areas as follows:

1. Alloy Development and Irradiation Performance
2. Plasma - Materials Interaction
3. Special Purpose Materials Development
4. Damage Analysis and Dosimetry
5. Radiation Source Development and Operation

The total DMFE materials program is funded at \$7.0 million in FY 1976. A booklet entitled "Programs of the Materials and Radiation Effects Branch", Division of Magnetic Fusion Energy is available on request.

1. Alloy Development and Irradiation Performance

FY 1976 Funding: \$1.8 Million

Contractors: HEDL, LLL, LASL, ORNL, PNL, Universities

The objective of the Alloy Development and Irradiation Performance area is to provide the materials development and application for those structural materials which are subject to significant radiation damage. The prime technical objective is the development of a structural material for the first wall and structural elements for the blanket and shield of a commercial fusion power reactor.

The scope of these activities includes (a) the definition of material requirements that are needed to satisfy fusion power system design goals including the Experimental Power Reactor (EPR), the Demonstration Plant (DEMO) and Commercial Fusion Reactors, (b) the evaluation of existing materials to meet those goals, (c) the development of new materials where they are required to achieve those goals, and (d) the development of the materials radiation data base required for design, construction and operation of fusion power systems.

2. Plasma-Materials Interaction

FY 1976 Funding \$1.7 Million

Contractors: ANL, GA, ORNL, PNL, Sandia, Universities

One of the major objectives of the Plasma-Materials Interaction area is to support the Alloy Development and Irradiation Performance task. The scope of Plasma-Materials Interactions, however, is much broader since the impact of this technical area is very important in near-term confinement systems, such as tokamaks. The approach is to treat the plasma-wall interaction as an integral problem covering both the effects on the plasma as well as the effects on the first wall.

Specific objectives are:

- To treat surface effects from the standpoints of plasma contamination, wall erosion, and device efficiency
- To contribute to the solution of problems in confinement experiments over the near term while developing the data base for dealing with possibly more severe problems in reactors
- To evaluate surfaces as part of a completely integrated system consisting of the plasma and its perimeter, the external blanket, subsystems for vacuum pumping, fueling and ash removal, and possibly bumpers or divertors

- To develop new materials resistant to surface damage in concert with overall alloy development tasks for commercial fusion power.

3. Special Purpose Materials Development

FY 1976 Funding: \$0.8 Million

Contractors: ANL, BNL, LASL, ORNL, PNL

This technical area covers the development of materials other than first wall/structural materials described previously. Included are the following materials applications:

- Advanced superconductors
- Structural materials for superconducting magnet support
- Insulators for superconducting magnets
- Insulators for structural applications such as the theta pinch first wall
- Insulators for components such as neutral beams
- Moderator and breeding materials
- Materials for heat transfer systems and power-conversion (secondary) systems
- Others as they become identified

4. Damage Analysis and Dosimetry

FY 1976 Funding: \$0.9 Million

Contractors: AI, ANL, BNL, HEDL, LASL, ORNL, Universities

The objectives of the Damage Analysis and Dosimetry area are to characterize available irradiation test environments and to establish a basis for predicting materials performance under irradiation in a fusion reactor environment. This will be accomplished by materials irradiation data obtained in fission reactors, accelerator based neutron test environments and charged particle irradiations.

The scope of the Damage Analysis and Dosimetry area includes development and application of the methodology and nuclear data base required for characterization of the neutronic and damage parameters of the reactor and test environments, the development of fundamental radiation damage models, the development of methods to account for interactive phenomena in the evaluation of damage

structures, the relationship of structure and damage parameters to property changes, and the application of the above to the prediction of material performance in fusion reactor systems.

5. Radiation Source Development and Operation

FY 1976 Funding: \$1.8 Million

Contractors: ANL, BNL, LASL, LLL, MIT

The objectives of this area are to define the radiation environment of fusion reactors and to pursue the development of neutron and plasma sources to simulate this environment for materials testing. Since fusion reactors are not now available for testing, high energy neutron and plasma sources are needed to develop materials for commercial fusion power.

High energy neutron sources are based on the deuterium-tritium (DT) reaction to produce 14 MeV neutrons and on the Li(D,n) and Be(D,n) stripping reactions to produce a broad neutron spectrum of high energy. Two DT neutron sources authorized for construction by Congress are the Rotating Target Neutron Source, RTNS, being built at LLL and the Intense Neutron Source, INS, being built at LASL. The RTNS is designed for 14 MeV neutron fluxes on the order of $2 \times 10^{13} \text{ n/cm}^2\text{-sec}$ at the specimen, while the INS is designed for 14 MeV fluxes of up to $10^{14} \text{ n/cm}^2\text{-sec}$ at the specimen. Proposals for a Li(D,n) neutron source of much larger volume and higher flux are under evaluation. This source will have a distributed spectrum which has been shown in the past year to be an excellent simulation of the fusion reactor damage spectrum.

Fission reactors are used in the program for testing of nickel-bearing materials because of the two stage reaction for helium production in mixed fast and thermal reactor spectra. Thus, fission reactors permit simulation of helium/dpa damage accumulation similar to that which occurs in fusion reactors. Unfortunately, this statement is true only for nickel-bearing alloys, and high energy neutron sources are needed for all other materials.

The above sources will be used to accumulate neutron radiation data for materials development as well as to contribute to an understanding of damage analysis and extrapolation of fission reactor data to the high energy fusion reactor spectra.

Title: Blended Cements (Energy Conservation in Cement Through Waste Use)

Contractor: National Bureau of Standards
Washington, D. C.

Principal Investigator: Dr. James Clifton
(301-921-3275)

FY 1976 Funding: \$100K (FY 1976 Start)

Objective and Scope

To reduce the energy consumed in the manufacture of cement by blending fly-ash or slag with kiln-fired cement.

Commercial acceptance of blended cements would significantly reduce the energy consumed in the manufacture of portland cement; the percent savings being approximately proportional to the percentage of additive material. To promote the greater utilization of blended cements, laboratory data are being developed to demonstrate their relative performance characteristics, including sulfate resistance, soundness and alkali-aggregates reactions. The resulting data will support development of industrial specification standards for blended cements.

Title: Effects of Alternate Fuels on Refractories and Refractory Insulation

Contractor: Oak Ridge National Laboratory
Oak Ridge, Tennessee

Principal Investigator: Dr. V. J. Tennery
(615-483-1670)

FY 1976 Funding: \$140K (FY 1976 Start)

Objective and Scope

To provide industry with technical data for selection and operation of refractories resistant to the corrosive effects of alternate fuels (e.g. oils and coal).

Potentially detrimental effects of alternate fuels on generic classes of refractories are being identified and compared with available industrial and literature data (Sub Task A). Selected refractory system - alternate fuel screening tests will be conducted to verify and supplement available data. Based on these results, refractory - alternate fuel tests will be conducted under field conditions under industrial operating conditions. Results obtained from this project will identify the magnitude of problems with different classes of refractories, provide guidance for materials selection and operational limits for furnace operations, and, to identify R&D targets for improved refractory materials, combustion systems or fuels.

Title: Assessment of Industrial Thermal Insulation Technology

Contractor: Oak Ridge National Laboratory
Oak Ridge, Tennessee

Principal Investigator: Ralph G. Donnelly
(615-483-1265)

FY 1976 Funding: \$50K (FY 1976 Start)

Objective and Scope

To assess the status of industrial thermal insulation technology including suitability of materials, availability of property data, and evaluation of testing procedures.

The assessment was based on the sum inputs of insulation manufacturers and users, system designers, installers, consultants and measurement laboratories. Conclusions and recommendations were formulated in the following areas:

- o Available insulation materials
- o Energy conservation applications for insulation
- o Economics of insulation
- o Quality and quantity of thermal data, and,
- o Methods for determining thermal properties

A final report was published in March, 1976: Industrial Thermal Insulation - An Assessment, ORNL/TM-5283.

Title: Characterization of Paper Pulp Fibers

Contractor: National Bureau of Standards
Washington, D. C.

Principal Investigator: Dr. Edward L. Graminski
(301-921-3275)

FY 1976 Funding: \$120K (FY 1976 Start)

Objective and Scope:

To reduce the energy consumed in the production of paper through development of optical imaging methods for rapid characterization of pulp fibers.

The availability of a rapid method for classification of pulp fibers would promote increased utilization of recycled paper and would permit more efficient processing of virgin stock. For the development of optical-imaging methods, laboratory correlations of fiber optical characteristics and mechanical properties of papers will be determined. Mathematical algorithmics for the optical characteristics of fibers will be developed, and used for the development of on-line instrumentation.

Title: Conservation in the Lime and Cement Industry by Steam
Calcination

Contractor: Brookhaven National Laboratory
Upton, L.I., New York

Principal Investigator: Dr. Meyer Steinberg
(516-664-3036)

FY 1976 Funding: \$50K (FY 1976 Start)

Objective and Scope

To reduce the energy consumption for calcinating lime and cement by about 25 percent through the use of steam catalysis.

Prior experimentation has shown that steam can reduce the calcination temperatures for lime and cement by about 200 C° and results in more active products. Laboratory experiments will be performed to identify the process parameters of steam - enhanced calcination and to evaluate the relative reactivities of the products. The laboratory data will be used as the basis for pilot-plant studies and leading finally 8 full-scale demonstrations.

